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Review of RAAF Procedures for Qualifying Bonded Repair Technicians

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Air Vehicles Division

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Defence Science and Technology Organisation

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ABSTRACT

DSTO has recently undertaken a review of procedures employed by the Royal Australian Air Force (RAAF) Bonded Structures and Testing Team (BSTT) during testing and requalification of technicians who undertake bonded repairs on ADF aircraft. Currently, technicians are required to produce a bonded wedge test under examination from BSTT staff and, based on adherence to the current RAAF Engineering Standard, will be considered competent to undertake bonded repairs. From December 2001 to January 2004 a notable deterioration in the quality of the bonded wedge tests produced through qualification testing was observed and an audit of processes was undertaken to determine if any areas in the bonding and requalification testing may have been leading to the deterioration in quality. Based on an audit of the processes and subsequent experimental testing at DSTO, recommendations on improvements to bonding procedures have been made.

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Review of RAAF Procedures for Qualifying Bonded Repair Technicians

Executive Summary

The Bonded Structures Testing Team (BSTT) at the Royal Australian Air Force (RAAF) base Amberley are responsible for training and qualifying all technicians who perform bonded repairs on Australian Defence Force (ADF) aircraft using RAAF engineering standard DEFAUST 9005-A and the associated document AAP 7021.016-2. Qualification of bonding technicians involves the technician performing a wedge test under supervision from BSTT staff, who observe each of the steps in the surface treatment of the Al-2024T3 clad plates bonded with FM-300 adhesive. In the early part of 2003 it became apparent that although a number of technicians were correctly following the procedures detailed in AAP 7021.016-2, the wedge test results were not passing examination. They were exceeding the crack growth and cohesive failure limits specified in the standard. BSTT and ASI requested that DSTO investigate the bonding procedures employed during qualification to determine if there was a discernible cause for the deterioration in the wedge test results. DSTO staff have undertaken a number of investigations since October 2003. The investigations have included a visit to the BSTT bonding facility to review procedures and infrastructure. Additionally, post-failure examination of available wedge test specimens from BSTT qualification testing was carried out from December 2001 to January 2004.

This report details the findings of the investigations of the BSTT bonding operations and wedge samples from BSTT qualification testing and provides a number of recommendations that will assist in improving the quality control of the current surface treatment procedures being employed. It is anticipated that recommendations will be incorporated in the RAAF engineering standard and, together with development of statistical analysis procedures, should provide a more robust system for maintaining the quality of bonded repairs performed by the RAAF and other areas of defence. It is also anticipated that implementation of ongoing statistical analysis of wedge test qualification will provide rapid feedback for RAAF on any deviations from optimum performance and enable short response times to remedy problems that can develop in bonding operations.

Amongst the recommendations of the report are the implementation of a quality control tool that monitors wedge test quality and provides a plot of wedge test results with time, the use of a gloss meter to measure grit-blast quality objectively and the use of epoxy silane kits to reduce the risks of contamination and incorrect mixing ratios of chemicals during surface treatment.

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Andrew Rider joined DSTO in 1988. He has a PhD in Physical Chemistry from the University of New South Wales and is currently employed as a Senior Research Scientist with Air Vehicles Division. He is involved in research to support RAAF maintenance of bonded structure and application of composite repairs used on ADF aircraft.

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Roger Vodicka graduated BSc. (Hons.) from Monash University and joined DSTO in 1990. He currently works in the area of life cycle costing, assessing the main cost drivers in maintenance of current fleets and estimating costs associated with future acquisitions.

Gary Mathys
Maritime Platforms Division

Gary Mathys graduated with a Bachelor of Applied Science from Deakin University in 1980. He also has completed an Associate Diploma of Engineering (Plastics) at the Royal Melbourne Institute of Technology. In 1980 he joined the Platforms Sciences Laboratory and is involved in the characterisation of organic materials of defence significance. He currently is managing the molecular spectroscopy (infrared, raman and UV-Visible spectroscopy) and thermal analysis facility.

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Ivan Stoyanovski currently is a Senior Technician in the Composites Group within Air Vehicles Division. He has extensive experience in composite manufacturing and has applied several bonded composite repairs to military aircraft.

Contents

Glossary

Abbreviation	Meaning
BSTT	Bonded Structures Testing Team, Training team with the responsibility of training and requalifying technicians who undertake bonded repairs on ADF aircraft.
ASI	Aircraft Structural Integrity Section at the Directorate General of Technical Airworthiness, Royal Australian Air Force
Adhesion Failure	Level of failure visually observed which appears to occur at the interface between the adhesive and the substrate
Cohesion Failure	Level of failure visually observed which appears to occur within the adhesive layer
Milli-Q water	Trademark name for water which has been distilled and deionised and is measured to have a resistance of 18 $Mohm.cm^{-1}$
XPS	X-ray photoelectron spectroscopy. A surface sensitive technique that can be used to measure the atomic concentration of elements in the outer 10nm of solid surfaces
RAIR	Reflection-Absorption Infrared Spectroscopy. An IR technique in which the incident IR radiation is directed at a grazing incidence on a solid surface and the reflected beam is measured to determine the surface species present based on characteristic vibration of chemical species in the mid-IR region.
Transmission IR	IR spectroscopy in which the incident IR beam passes through the sample being analysed.
IR Microscopy	Infrared spectra are acquired from localised areas using an optical microscope that focuses the infrared beam in a localise area and specialised optics allow the reflected light to be analysed in the FT-IR spectrometer.
FM300	Cytec structural epoxy adhesive with a cure performed at 177^{o}C for 1 hour
FM300-2K	A modified version of FM300, with a cure performed at 120oC for 90 minutes. Similar mechanical properties to FM300, but reduced cure temperature makes it more suitable for field use.
ISO	ISO (International Standards Organization) is the International Standards Organizations. They do not create standards but (as with ANSI) provide a means of verifying that a proposed standard has met certain requirements for due process, consensus, and other criteria by those developing the standard
DIN	Deutsches Institut für Normung (DIN, the German Institute for Standardization) is a German national organization for standardization. DIN and mini-DIN connectors are familiar to computer users all over the world, but DIN issues standards in any conceivable area
ASTM	American Society for Testing and Materials. An organization to establish test standards for materials, products, systems, and services for a wide range of industries

1. Introduction

The Bonded Structures Testing Team (BSTT) at the Royal Australian Air Force (RAAF) base Amberley are responsible for training and qualifying all technicians who perform bonded repairs on ADF aircraft using RAAF engineering standard DEFAUST 9005-A [1] and the associated document AAP 7021.016-2 [2]. Qualification of bonding technicians involves performing a wedge test under supervision from BSTT staff, who observe each of the steps involved in the surface treatment of the Al-2024T3 clad plates bonded with FM-300 adhesive. In the early part of 2003 it became apparent that a number of technicians were correctly following the procedures detailed in AAP 7021.016-2, however, the wedge test results were not passing, based on the crack growth and cohesive failure limits specified in the standard. BSTT and ASI requested that DSTO investigate the bonding procedures employed during qualification to determine if there was a discernible cause for the deterioration in the wedge test results. DSTO staff have undertaken a number of investigations since October 2003. The investigations have included a visit to the BSTT bonding facility to review procedures and infrastructure. Additionally, post -failure examination of available wedge test specimens from BSTT qualification testing was carried out from December 2001 to January 2004.

The following report details the findings of the investigations of the BSTT bonding operations and wedge samples from BSTT qualification testing and provides a number of recommendations that will assist in improving the quality control of the current surface treatment procedures being employed. It is anticipated that recommendations be incorporated in the RAAF engineering standard and, together with development of statistical analysis procedures, should provide a more robust system for maintaining the quality of bonded repairs performed by RAAF and other areas of defence. It is also anticipated that implementation of ongoing statistical analysis of wedge test qualification will provide rapid feedback for RAAF on any deviations from optimum performance and enable short response times to remedy problems that can develop in the bonding operations.

2. Surface treatment and wedge testing

2.1 Surface treatment

The recommended surface treatment for epoxy adhesive bonding to aluminium is detailed in AAP 7021.016-2 [2]. The procedure involves the following:

1) solvent wiping: single wiping of the aluminium surface using methyl ethyl ketone (MEK) soaked lanoline and lint free tissues. A fresh tissue is used after each pass. Single wiping is conducted along the grain direction and at 90^0 relative to the grain until no observable debris or staining of the tissue can be observed,

2) Scotchbrite® abrasion with MEK: following solvent wiping the surface is abraded with the Scotchbrite® pad soaked in MEK along the grain direction and at 90^0 relative to the grain until a uniform surface appearance is observed. Single wiping of the aluminium surface then uses MEK soaked lanoline and lint free tissues. A fresh tissue is used after each pass. Wiping is conducted in the direction of the abrasion until no presence of debris or staining of the tissue can be observed.
3) Scotchbrite® abrasion with deionised water: following step 2, the surface is abraded with the Scotchbrite® pad soaked in deionised water along the grain direction and at 90^0 relative to the grain until a uniform surface appearance is observed. Single wiping of the aluminium surface then uses deionised water soaked lanoline and lint free tissues. A fresh tissue is used after each pass. Wiping is conducted in the direction of the abrasion until no presence of debris or staining of the tissue can be observed.
4) Water-break testing: The surface is water-break tested by thoroughly wetting the surface prepared in 3) with deionised water and observing that no areas are free of water. The surface is then gradually dried using a hot air gun and moisture should evaporate in a uniform manner without any water-breaks. If water-break areas are present steps 1)-3) must be repeated.
5) Drying: The surface is dried in an oven at 110^0C for 5 minutes prior to grit-blasting,
6) Grit-blasting: uniform grit-blasting of the surface employs 50 μm alumina grit and dry nitrogen propellant with a pressure of 450kPa and a working distance of 15 to 20cm
7) Silane treatment: a 1% aqueous solution of γ-glycidoxypropyltrimethoxysilane (γ-GPS) is stirred for 1 hour prior to commencing the surface pre-treatment steps listed above. Distilled water is used to prepare the silane solution. The grit-blasted aluminium surface is "immersed" in the silane solution for 15 minutes by applying the solution regularly to the aluminium surface from clean lanoline and lint free tissues. The surface is then allowed to drain free of excess solution, followed by drying in an 110^0C oven for 60 minutes.

2.2 Wedge test preparation and testing

Wedge tests are constructed from 0.125 inch Al-2024 T3 clad alloy using FM300 adhesive in a configuration shown in Figure 1 [2]. FM300 is placed between two plates prepared using the method described in section 2.1 and cured at 177°C for 90 minutes using a ramp rate of 3°C per minute. The vacuum of 30 inches of mercury is reduced to 10 inches of mercury when the heat-up temperature reaches 130°C.

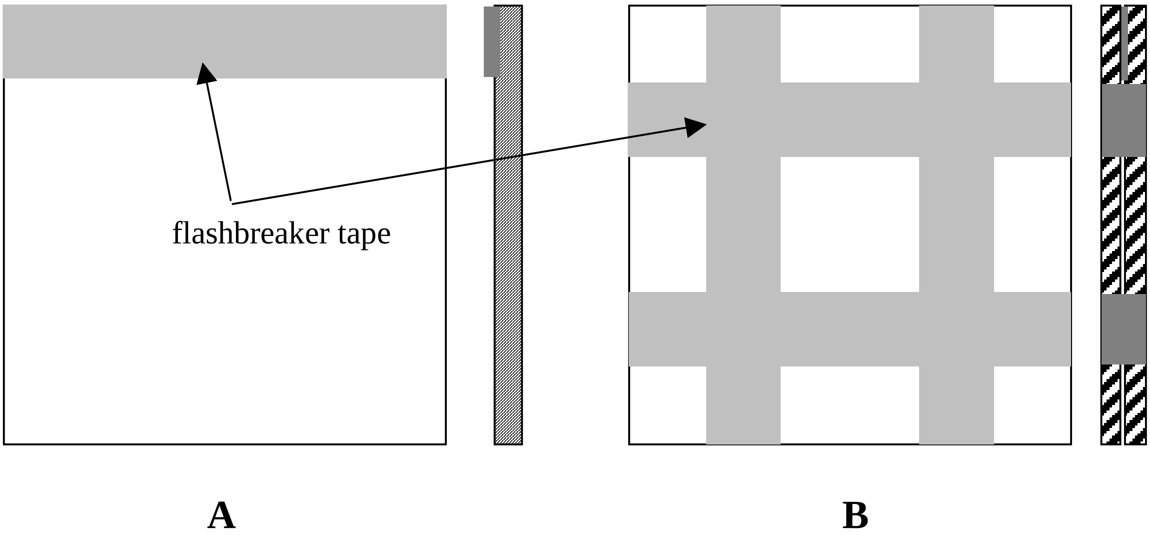

Figure 1 Placement of flashbreaker tape to enable (A) wedge insertion and (B) to prevent slippage during curing of the bonded wedge test[2].

Figure 2 indicates how the plates are cut into five specimens and the position that the wedge is inserted prior to placement of the sample in the humid environment. Initially, the sample is allowed to equilibrate at room temperature in the laboratory environment prior to insertion in the humid test chamber.

The allowable crack growth in the dry and humid environment over 24 and 48 hours is 42mm and 48.5mm, respectively. Additionally, the region of crack growth cannot exhibit less than 90% cohesion failure in any of the 4 specimens that record the lowest crack growth, for the technician to be qualified.

Cohesion failure refers to failure where fracture propagates within the adhesive layer. In contrast adhesion failure refers to failure where the crack propagates at the interface between the adhesive and aluminium layers.

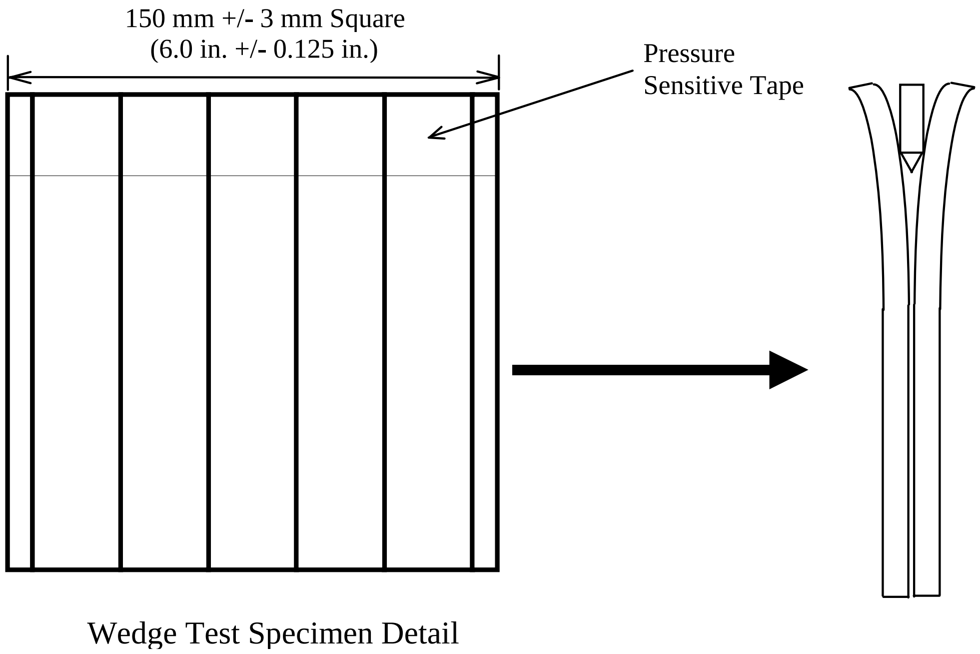

Figure 2 Diagram indicating the dimensions of the specimens cut from the bonded plates and the positioning of the wedge before insertion in the 50^{o}C, 100% R.H. [2].

2.3 Failure analysis and wedge test inspection

2.3.1 Wedge Test Inspection and Data Collection

All available wedge specimens and associated documentation produced as a result of BSTT requalification testing was examined at DSTO. Details of data collected and a description explaining the significance of the data and how it was collected is provided in Table 1. All data has been consolidated in an Excel spreadsheet to enable further data recording and development of statistical analysis that will form part of a quality control system for future wedge qualification.

Table 1 The data collected as a part of testing and examination of the wedge tests produced through BSTT training and requalification

Data Description	**Mode of Collection**	**Significance**
Sample No	Data from BSTT	Identification of sample by technician name
Adhesive	Data from BSTT and wedge inspection	Adhesive type

Adherends	RAAF Standard and wedge inspection	Metal and alloy type
Exposure	RAAF Standard	Environment used for wedge testing (50°C and 100% R.H.)
Surface Prep	RAAF Standard	Details exact steps used in surface preparation of adherend and if any deviations from standard have occurred
Test Date (ddd/mmm/yy)	Recorded by DSTO during wedge test	Date when test starts and finshes
Initial crack-length (mm)	Recorded by DSTO during wedge test	Provides initial crack-length of each wedge test specimen from number 1 to 5 after 1 hour of crack-growth in the laboratory environment
24 hour crack-length (mm)	Recorded by DSTO during wedge test	Provides 24 hour crack-length of each wedge test specimen from number 1 to 5 after 24 hours of crack-growth in the humid environment
48 hour crack-length (mm)	Recorded by DSTO during wedge test	Provides 48 hour crack-length of each wedge test specimen from number 1 to 5 after 48 hours of crack-growth in the humid environment
Mean initial crack-length (mm)	Recorded by DSTO during wedge test	Provides mean initial crack-length
Mean 24 hour crack-length (mm)	Recorded by DSTO during wedge test	Provides mean 24 hour crack-length
Mean 48 hour crack-length (mm)	Recorded by DSTO during wedge test	Provides mean 48 hour crack-length
Requalification date (ddd/mmm/yy)	Data from BSTT	Date when wedge samples made and requalification testing was carried out
Batch No./ Roll No.	Data from BSTT	Batch and roll number of adhesive used for testing
Adhesive arrival date (ddd/mmm/yy)	Data from BSTT	Date adhesive batch arrives at Amberley
Adhesive requalification date (ddd/mmm/yy)	Data from BSTT	Date adhesive batch is relifed to extend manufacturer's use by date by 6 months
Adhesive expiry date (ddd/mmm/yy)	Data from BSTT	Date adhesive batch exceeds use by date

Silane Batch No.	Data from BSTT	Epoxy silane coupling agent batch number from manufacturer
Water break drying temperature	Data from BSTT	Temperature metal plates dried at after water break testing
Silane Drying Temperature	Data from BSTT	Temperature metal plates dried at after being treated with the 1% epoxy silane solution
Temperature (^{o}C)	Data from BSTT	Temperature in Bonding facility during wedge test manufacturing
Humidity (%)	Data from BSTT	Relative Humidity in Bonding facility during wedge test manufacturing
Cohesion (%)	Recorded by DSTO during wedge test	Percentage of cohesion failure observed visually on the fracture surfaces of the wedge test specimens from number 1 to 5 by technician examining failure surfaces
Grouping	Recorded by DSTO during wedge test	The group number based on the date of receipt and testing of wedge samples from BSTT at DSTO.
Supervisor	Data from BSTT	Name of the BSTT Supervisor examining the technicians during wedge test requalification
Unit	Data from BSTT	Location of company or unit of technician being tested
Organisation	Data from BSTT	Affiliation of technician
Course	Data from BSTT	Course number attended by technician for training prior to wedge qualification
Service No.	Data from BSTT	Service number of technician be tested
Test Site	Data from BSTT	Location at which the testing occurred
Name	Data from BSTT	Full name and rank (if applicable) of technician being tested
Pass/Fail	Recorded by DSTO during wedge test	Pass or fail of the wedge samples as determined by the technician testing BSTT specimens
Grit-blast level	Recorded by DSTO during wedge test	Assessment of the quality of the grit-blast based on 10X digital image acquired from non-bonded area of wedge sample
Large Void (%)	Recorded by DSTO during wedge test	Provides visual assessment of area of each wedge test specimen from number 1 to 5 containing largely voided regions
Small Voids (%)	Recorded by DSTO during wedge test	Provides visual assessment of area of each wedge test specimen from number 1 to 5 containing sub-millimetre voided regions

Adhesion Failure[1] (%)	Recorded by DSTO during wedge test	Percentage of adhesion failure observed visually on the fracture surfaces of the wedge test specimens from number 1 to 5 by DSTO scientist examining failure surfaces
Comments	Recorded by DSTO during wedge test	Unique elements of wedge test sample or group that distinguish or clarify results

2.3.2 Grit-blast surface inspection

All wedge samples recovered from BSTT were photographed using a Cannon EOS D60 digital camera with a 100mm macro lens, operating with aperture priority (AE) mode and an aperture setting of 16 to provide sufficient depth of field for the grit-blasted images being examined. Exposure time, using the natural fluorescent lighting of the laboratory, was typically 6 seconds.

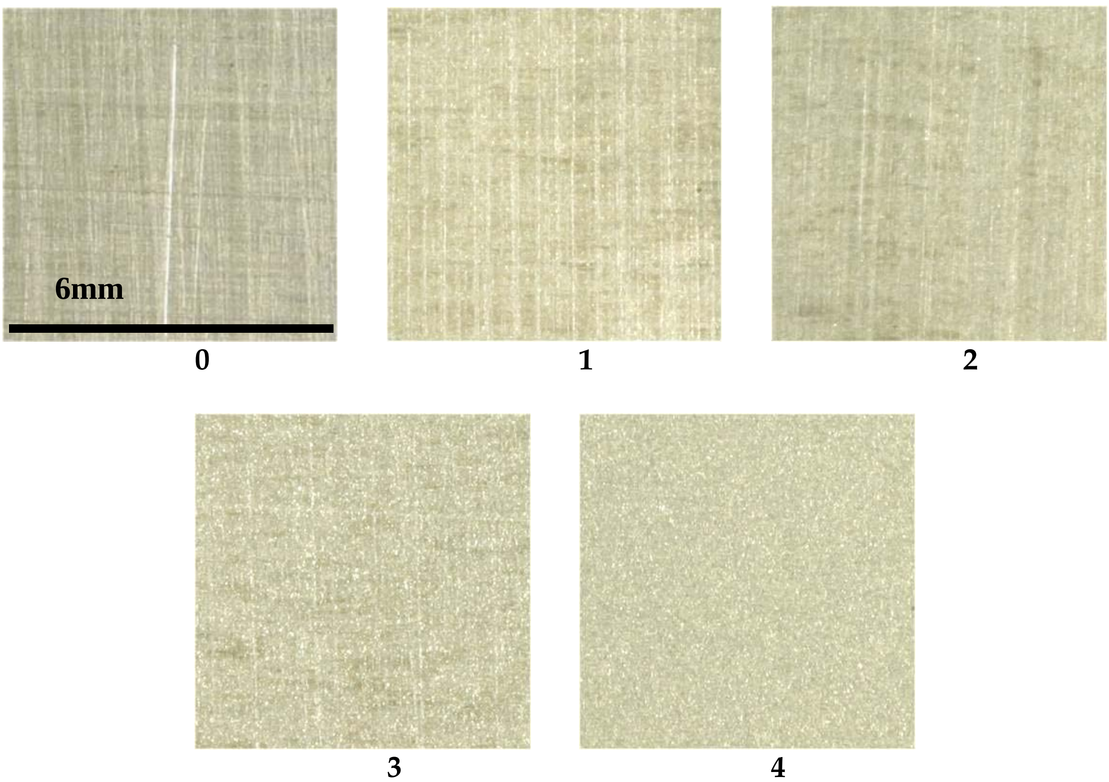

Figure 3 Grit-blast images indicating the scale used to grade the quality of grit-blast achieved on the BSTT requalification wedge test samples, where 0 is poor and 4 is satisfactory

[1] Refer to Glossary for description of Adhesion and Cohesion failure.

Figure 3 indicates the range in grit-blast quality and the arbitrary ranking scheme employed to classify the quality of the grit-blasted surfaces. In the case of a "0" ranking, evidence of grit-blasting was absent and the abrasion marks are clear. As the ranking number increases to "4" the scratches from abrasion disappear and the speckled surface resulting from alumina grit particles that have embedded in the aluminium surface becomes dominant. Images are taken from a 6mm x 6mm area.

2.3.3 Surface analysis of failed BSTT wedge samples

Selected wedge test samples that had not passed the RAAF standard and exhibited significant areas of adhesion[1] failure in the humid environment were analysed using X-ray photoelectron spectroscopy (XPS). XPS was carried out using a Kratos XSAM-800 using Mg K_{α} X-rays at 15kV and 15mA in fixed retard ratio and high magnification mode, which provided an analysis area of approximately 2mm in diameter. Quantification was performed using the original manufacturer's sensitivity factors. XPS is a surface sensitive technique that can provide an estimate of the elemental concentrations of the outer surface of solids. The elemental concentrations can be used to estimate the locus of fracture, given the different components of the bondline have unique elements and chemical concentrations.

2.4 Quality control

2.4.1 Quality control of grit-blasting

Commercially available equipment that has the potential to measure variations in grit-blast treatment prior to bonding was trialed. IDM Instruments [3] demonstrated a ColorQA version 2 handheld colour comparison device used for portable colour measurements in factory and field situations. The unit retails at $625.35. Eight samples with extreme ranges of grit-blasting and abrasion with Scotchbrite® pads were examined. Four samples of clad Al-2024T3 received heavy abrasion prior to a range of grit-blast treatments ranging from light to heavy. Four additional samples received light abrasion prior to a range of grit-blast treatments ranging from light to heavy. Colour measurements at five locations on each sample were taken to determine detection sensitivity of the unit.

A gloss-meter unit from BYK Gardner [4], demonstrated by IDM Instruments [3], was also examined at DSTO on the same samples investigated with the colour meter. The Hand Held Micro TRI Gloss® can take gloss measurements at three angles (20^{o}, 65^{o} and 80^{o}), is self-calibrating and can download measurements to a spreadsheet application. Measurements comply with ISO, DIN and ASTM standards (refer to the Glossary for definitions of the different standards).

2.4.2 Fourier transform infra-red analysis of epoxy silane

Aluminium treated with a 1% aqueous solution of epoxy silane (γ-GPS) was analysed using Reflection-Absorption Infrared[2] (RAIR). Spectra were collected using a grazing angle bench that enabled the beam to be focussed on the sample at 80^0 relative to the surface normal. The reflected beam was recorded with a $4cm^{-1}$ resolution and averaged over 256 scans. Transmission infrared[2] spectra of neat γ-GPS were also recorded using a long path length cell. γ-GPS was analysed from three batches. A batch received from BSTT Amberley that was being used in current wedge test qualifications, a fresh sample purchased from Sigma-Aldrich chemical company and an aged sample that was at least 6 years old and hand been stored in a laboratory environment.

RAIR studies required that the Al-2024 T3 clad aluminium sample be polished metallographically to a 1µm finish, followed by treatment for 15 minutes in a 65^oC chromic acid etch (CAE) solution, followed by tap and de-ionised water rinsing. The CAE solution had the following composition (g dm^{-3}): $Na_2Cr_2O_7.2H2O$ (60), H_2SO_4 (318), Al-2024 T3 aluminium (1.3), de-ionised water (balance) [5]. Following rinsing the sample was dipped in the 1% aqueous γ-GPS solution for 10 minutes, removed and blown dry with a stream of high purity nitrogen and analysed in the spectrometer. A CAE treated Al-2024 T3 clad aluminium sample was also analysed to provide the spectrum that was used for background subtraction.

2.4.3 Epoxy silane kits for transport, control and improved sensitivity

Kits containing epoxy silane, 0.1% acetic acid and distilled deionised water were prepared to determine if RAAF could implement a central store for dispensing epoxy silane in a form that would improve traceability of the chemicals used in the process. It was also hoped that the kits would reduce the amount of equipment and material needed to be transported by BSTT when travelling to ADF bases for requalification testing. Acetic acid was incorporated into the kits to ensure the pH of the silane solution was acidic and, therefore, optimised for hydrolysis of the silane. Hydrolysis is critical for successful deposition of the silane films as it ensures that silanol groups can form and then react with the metal substrate and/or cross-link to form a hydrolytically stable film. Experiments involved cleaning and the measuring out of epoxy-silane, acetic acid and distilled and deionised water into Nalgene containers as detailed below:

Water-Nalgene® 500mL Wide Mouth HDPE Bottles (Catalogue No. 21040016)

- Rinse with AR grade MEK (attach lid and shake)
- Rinse with distilled water (attach lid and shake)
- Fill with boiling distilled water and empty (attach lid and shake)

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[2] Refer Glossary for definition of RAIR and transmission IR.

- Fill with Milli-Q[3] water (490mL)

Epoxy Silane-Nalgene® 15mL LDPE Dropper Bottles (Catalogue No. 21040016)

- Rinse with AR grade MEK
- Rinse with distilled water
- Fill with 80°C distilled water and empty
- Dry in oven at 70°C (upside down to sure all moisture drains) for 1 hour
- Allow to cool to room temperature
- Pipette out 5g of epoxy silane into the dropper bottle using a micro-balance.

0.1% Acetic Acid-Nalgene® 15mL LDPE Dropper Bottles (Catalogue No. 21040016)

- In a clean 2L beaker, measure out 1L of Milli-Q water
- Add 50g of Glacial Acetic Acid and mix using magnetic stirrer
- Pipette out 10mL into the cleaned dropper bottles using a micro-balance.

Aging Studies of Nalgene Containers

- Place water, epoxy silane and acetic acid containers in oven at 50°C and remove at 1 week, 1 month, 3 months, 6 months and 12 months and produce a wedge test using the grit-blast and silane process (sec. 2.1) and FM-300 adhesive. Also analyse epoxy silane using the techniques detailed in section 2.4.2.

Additionally, a kit of epoxy silane was posted to BSTT for trial. Feedback regarding use of the kit was provided to DSTO and a wedge test specimen fabricated at BSTT using the kit was tested to verify laboratory results.

2.5 BSTT bonding facility site visit by DSTO

Ivan Stoyanovski from DSTO visited BSTT at RAAF base Amberley to review facilities and procedures at the bonding facility. The major objectives of the visit were to:

1) identify any variations in the bonding procedures conducted at BSTT with those at DSTO
2) assess the bonding facilities and storage and identify any variations from specifications detailed in AAP 7021.016-2
3) determine if any significant changes in processes or materials may have occurred around the period when wedge test failures increased
4) manufacture a wedge test alongside a BSTT instructor and examine results.
5) Collect samples of materials used in the bonding operations and analyse for potential contaminants at DSTO

[3] Refer to Glossary for definition of Milli-Q water

2.6 Surface treatment sensitivity studies

A series of wedge tests were manufactured at DSTO, Melbourne, to examine the influence of extreme deviations in process and the crack-growth levels expected. The wedge tests examined the influence of silicone contaminant in the bonding environment, absence of grit-blast, Scotcbrite abrasion and detritus removal after abrasion on crack growth and failure modes. The results are intended to provide an indication of which steps in the bonding process have the biggest effect on crack growth and failure mode. All wedges were manufactured using Al-2024T3 clad aluminium and FM-300.

3. Results

3.1 BSTT qualification- wedge test inspection

Table 2 provides a summary of the dates and pass/fail rates for each group tested at BSTT between December 2001 and November 2003. The major features of the data are the high failure rates observed for group 14 and groups 5, 17, 18 and 19. Apart from group 14, FM-300-2K[4] was used. FM-300-2K is not typically employed by RAAF for the wedge requalifications, although group 4 used FM-300-2K due to a shortage of FM-300 during a 1 month period between July and August of 2002. Groups 17 to 19 used FM-300-2K due to concerns over the poor results observed for FM-300 in group 14. Apart from group 14, groups 1, 5, 8, 11 and 20 had failures when FM-300 was used.

Table 2 Summary of wedge test data from BSTT qualification testing from December 2001 to November 2003

Gp	No.	Date	Course no./ Requal	Site	Grit-blast quality (0-bad, 4-okay) Quality-No.	Adhes. batch	Pass/ Fail	Comment
1	5	19/12/2001	Course 1	Amberley	1-1, 2-2, 3-2	1066	3/2	Poor grit-blast on both fails
2	4	14/3/2002	Course 1	Amberley	4-4	1066	4/0	Good grit-blast
3	4	3/5/2002	Course 2	Amberley	4-4	1066	4/0	Good grit-blast
4	5	14/6/2002	Course 3	Amberley	4-4	1066	4/1	Failed sample missing

[4] FM300 and FM300-2K are defined in the Glossary

5	4	25/7/ 2002	Course 4	Amberley	3-3	N/A	1/3	FM300-2K used
6	4	5/9/2 002	Course 5	Amberley	4-4	1082	4/0	Good grit-blast
7	3	17/10 /2002	Course 6	Amberley	3-1 4-2	1082	3/0	grit-blast okay
8	4	31/10 /2002	requal	Amberley	0-1 3-1 4-2	1082	3/1	Bad grit-blast on failed sample
9	18	13-28 /11/ 2002	3 from Course 7, remainde r requal	Amberley	3-4 4-15	1082	19/0	3 tests marginal pass
10	2	15/1/ 2003	requal	Amberley	4-2	1082	2/0	Good grit-blast
11	3	27/2/ 2003	Course 8	Amberley	2-2 4-1	1082	1/2	poor grit-blast on failed samples
12	9	1-10 /4/ 2003	requal	Nowra and William-town	3-4 4-5	1082	9/0	Good group, very low adhesive failure
13	4	22-5- 2003	Course 9	Amberley	4-4	1082	4/0	Good grit-blast, low adhesion failure
14	13	11-18 /6/20 03	requal	Edinburgh	1-2 3-1 4-10	1082	6/7	2 failures associated with bad grit-blasting. 4 failures had good grit-blast Low voiding may suggest light abrade
15	4	16/7/ 2003	Course 10	Amberley	3-2 4-2	1082	4/0	Grit-blast okay, no adhesion failure
16	3	13/8/ 2003	requal	Tindal	3-3	1082?	3/0	1 result marginal pass, with high adhesion failure. Low voiding may suggest light abrade
17	3	15/9/ 2003	requal	Townsville	3-1 4-2	N/A	1/2	FM300-2K used
18	4	25/9/ 2003	Course 11	Amberley	4-4	N/A	1/3	FM300-2K used
19	6	21-22 /10/2 003	requal	Williamtow n	4-6	N/A	3/3	FM300-2K used

20a	2	17/11 /03	Inspection visit	Amberley	4-2	1112/ 0022A	2/0	New batch of FM-300, Ivan Stoyanovski and Dave Sowden during DSTO visit to assess operation
20	14	11-19 /11/2 003	Requal	Amberley	3-3 4-11	1112/ 0022A	13/1	1 failure and other marginal result both tested on the same day. Grit-blast okay, no obvious reason for failure
21	5	19/11 /2003	Course 12 and 1 requal	Amberley	3-1 4-4	1112/ 0022A	5/0	Grit-blast okay

Further analysis of the data indicates that FM-300 exhibited a failure rate of 13% compared to 65% when FM-300-2K adhesive was used (Table 3). Of the 14 failed samples that used FM-300, 50% of these had grit-blast treatments at levels of 2 or less (Figure 3). FM-300-2K samples did not indicate any obvious deficiencies in grit-blast treatment. Table 3 also indicates that, when FM-300 was used, there were fewer failures when testing was performed as a part of training course examination, compared to requalification of technicians. FM-300-2K showed no significant difference in requalification or course testing failure.

Table 3 Summary of Failure Rate and Grit-blast quality for BSTT Requalification testing

	FM-300	**FM-300-2K**
Tests	107	17
Failed (No.)	14	11
Failed (%)	13.1	64.7
Poor Grit-blast (No.)	7	0
Poor Grit-blast (%)	6.5	0
Poor Grit-blast associated with failed tests (%)	50	0
Failures after course	5	6
Failures after requalification	9	5

Failure rate for each site where wedge tests were manufactured is provided in Table 4 for FM-300 and FM-300-2, indicating poor results for Edinburgh when FM-300 was used. Pass rates for FM-300-2K are poor for all sites.

Table 4 Pass rates for FM-300 and FM-300-2K based on manufacturing location.

Location	FM-300			FM-300-2K		
	Pass	Fail	Pass (%)	Pass	Fail	Pass (%)
Amberley	75	7	91.5	2	6	25.0
Williamtown	7	0	100.0	3	3	50.0
Nowra	2	0	100.0	---	---	---
Edinburgh	6	7	46.2	---	---	---
Tindal	3	0	100.0	---	---	---
Townsville	---	---	---	1	2	33.3

Table 5 indicates the failure rates based on organisation and group classification. The high failure rate observed for 10 and 11 Squadron is due in part to the high failure rates observed for group 14 (Table 2), where the testing was performed at Edinburgh. It should be noted, however, that the other groups from RAO and 24 Squadron tested at the same time passed. For the two organisations performing the most number of requalification testing, Boeing Amberley and RAAF, the RAAF pass rate is almost 15% lower than Boeing. In the case of Army, 2 of the 3 failures are known to have been caused by bad grit-blasting and occurred when the same BSTT supervisor was present. The third failure cause is unknown as the specimen was unavailable. This essentially leaves the failures for RAAF the most difficult to explain, given, one of the Boeing Amberley failures can be attributed to poor grit-blasting.

Table 5 Failure rates for different groups and organisations tested during qualification by wedge test using FM-300.

Organ-isation	Group	FM-300			
		Pass	Fail	Pass(%)	Pass(%) excluding bad gb (no.)
RAAF	1 SQN	2	0	100	
	3SQN	5	0	100	
	6SQN	4	0	100	
	10SQN	4	4	50	67(2)
	11SQN	4	3	57	57(0)
	24SQN	1	0	100	
	37SQN	3	1	75	100(1)
	75SQN	4	0	100	
	77SQN	1	0	100	
	161SQN	2	0	100	
	162SQN	1	0	100	
	Amberley	1	0	100	
	BSTT	2	1	67	100(1)
	2OCU	2	0	100	
	RAO	2	0	100	
	TOTAL	**38**	**9**	**80.9**	**88.4**

Table 5 con't Failure rates for different groups and organisations tested during qualification by wedge test using FM-300.

Organ-isation	Group	Pass	Fail	Pass(%)	Pass(%) excluding bad gb (no.)
ARMY	1AVN	0	1	0	(1)
	5AVN	4	1	80	80(0)
	ASGW	5	1	83	?
	TOTAL	**9**	**3**	**75.0**	**?**
NAVY	815SQN	1	0	100	
	816SQN	2	0	100	
	TOTAL	**3**	**0**	**100.0**	
COMM.	Boeing Amberley	36	2	94.7	97.3(1)
	Helitech Brisbane	2	0	100	
	ASGW, Helitech	1	0	100	
	Hunter, Nowra	2	0	100	
	TOTAL	**41**	**2**	**95.3**	**97.6**

Failure rates for FM-300 as a function of batch and batch age are shown in Table 6 and Figure 4. Batches 1066 and 1082 show similar failure levels, with the new batch of 1112 being difficult to assess due to the limited adhesive life. The trend in failure rates as a function of batch age does not indicate any obvious similarities for the different batches.

Table 6 Failure percentages as a function of FM-300 batch number.

FM-300 Batch No.	Dates of Use	Total Age	Tests	Failures	Failure (%)
1066	19/12/01 to 14/6/02	8 months	18	3	16.7%
1082	5/9/02 to 13/8/03	12 months	68	10	14.7%
1112	17/11/03 to	1 month	21	1	4.8%

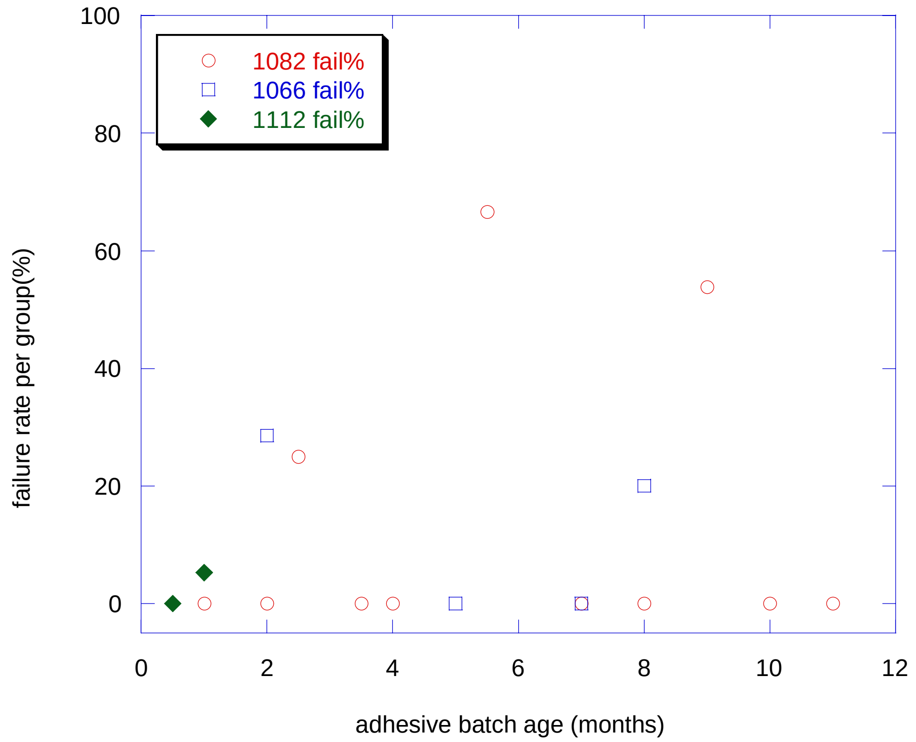

Figure 4 Failure percentage for BSTT wedge test requalification as a function of batch age for the 3 batches of FM-300 used to November 2004

3.2 Surface analysis of failed wedge samples

3.2.1 XPS analysis

Table 7 indicates the surface elemental composition of the failed samples from the BSTT testing. Details include the name of the technician, the group in which testing occurred, which of the 5 wedge specimens were analysed, the final crack-length of the sample and the size of the sample cut from the failed wedge piece. The "metal" and "adhesive" sides represent the failure surfaces that had a metallic and FM-300 appearance, respectively. Some indication of the locus of fracture can be determined on the basis that silicon is a constituent of the organosilane coupling agent, bromine is found in FM-300 adhesive, aluminium and high oxygen levels are typical of oxidised or hydrated aluminium, high carbon levels are typical of the FM-300 and sodium is often found on grit-blasted surfaces.

Table 7 XPS failure analysis of BSTT failed wedge samples indicating surface elemental concentrations on the "metal" and "adhesive" surfaces.

Name	Gp	Sp. No.	Final Crack (mm)	Size (mm)	Fail. Side	Atomic Concentration (%)						
						Al	Si	Br	C	N	O	Na
BW (gb 2)	11	1	49.9	25x10	Metal	11.5	---	0.4	51.7	2.0	34.4	---
				25x15	Adhes.	1.5	3.6	1.0	67.8	3.1	23.0	---
TB (gb 3)	11	2	41.2	25x6	Metal	11.9	---	1.0	44.4	2.0	40.3	0.5
				25x6	Adhes.	2.3	0.8	1.8	63.3	4.0	27.8	---
PV (gb 1)	14	4	47.2	25x15	Metal	11.6	---	0.5	46.9	1.8	38.6	0.6
				25x15	Adhes.	1.3	1.9	1.2	69.2	2.9	23.2	0.3
JL (gb 1)	14	5	47.4	14x12	Metal	13.3	---	0.8	41.6	2.1	42.2	0.1
				14x12	Adhes.	1.7	2.2	0.7	68.2	2.0	24.9	0.4
BS (gb-4)	14	4	49.2	25x12	Metal	16.5	---	1.0	32.7	1.7	46.8	0.5
				25x12	Adhes.	2.7	2.0	1.6	62.0	3.8	27.9	---
DT (gb-4)	14	5	50.9	15x12	Metal	17.2	---	0.8	29.1	1.6	50.6	0.6
				15x13	Adhes.	1.9	1.2	1.8	65.2	4.0	25.9	---
NS (gb-4)	14	5	51.5	14x12	Metal	14.9	---	1.1	34.8	2.0	46.1	1.0
				13x11	Adhes.	2.2	2.4	1.4	63.5	3.0	27.5	---
AP (gb 3)	14	1	51.7	20x13	Metal	13.3	---	1.0	37.0	2.1	45.8	0.8
				20x13	Adhes.	1.4	2.2	1.3	63.5	4.1	27.5	---
DR (gb 4)	14	5	53.4	17x15	Metal	15.4	---	0.9	35.5	1.7	46.1	0.4
				15x13	Adhes.	3.3	1.3	1.6	62.0	2.9	28.8	---
GW (gb-4)	20	1	55.1	15x8	Metal	14.9	---	0.7	38.1	1.5	44.0	0.8
				15x10	Adhes.	1.1	---	1.2	76.6	2.0	19.2	---

Comparing PV and JL (gb 1) with the other results in group 14 (gb 4), suggests that on the "metal" surface as the crack increases in length Al, O and Br levels increase and C levels decrease. A similar comparison on the "adhesive" surfaces suggests that as the crack increases in length Al, O, N and Br levels increase and C levels decrease. These trends may indicate that the crack propagates closer to the metal surface or into the oxide as the crack length increases and the bond becomes less durable. The increase in Br and N on the "adhesive" may suggest changes in adhesive failure surface that would be consistent with fracture closer to the interface where the adhesive may be less cured compared to the bulk Previous work looking at adhesive interfaces suggests that the epoxy cure reaction is reduced in the interface near aluminium surfaces [6]. The GW sample which was unique in that it was the only sample to fail in group 20 was found to differ on the "adhesive" failure surface, where C levels were notably higher than other samples and no Si was detected. The relatively high levels of carbon on the "metal" side of all samples does suggest that the crack may alternate between the interface and within the epoxy adhesive. The presence of sodium on both faces of some failure specimens may suggest that embedded grit is detected on both surfaces and that during fracture some of the grit is being removed from the aluminium as the adhesive separates from the metal substrate.

3.3 Quality control of metal surface treatments

3.3.1 Colour meter measurements for grit-blast severity

Table 8 indicates the colour measurements using the ColorQA version 2 handheld unit for the Scotchbrite® abraded and grit-blasted samples. Heavy, medium and light grit-blasted surfaces represent "4", "3" and "2", respectively, in Figure 3. All three colours show similar trends and for a heavily abraded surface there is a difference of approximately 30 units from 150 to 120 as the grit-blast approaches a standard level. There is a difference of approximately 70 units from 190 to 120 as the grit-blast approaches a standard level for surfaces that are abraded lightly.

Table 8 Red, green and blue colour values (0-255) for samples subjected to varying degrees of grit-blasting and Scotchbrite® abrasion.

Scotchbrite	**Grit-blast**	**Position**	**Red**	**Green**	**Blue**
Heavy	None	1	146	143	139
		2	158	160	153
		3	157	157	150
		4	144	142	137
		5	148	148	139
		Average	**151**	**150**	**144**
Heavy	Light	1	134	136	129
		2	150	157	148
		3	148	153	145
		4	139	139	134
		5	157	166	153
		Average	**146**	**150**	**142**
Heavy	Medium-Light	1	136	137	132
		2	138	139	133
		3	133	135	128
		4	152	156	148
		5	149	154	147
		Average	**142**	**144**	**138**
Heavy	Medium	1	118	122	117
		2	119	122	118
		3	126	126	121
		4	118	123	115
		5	112	117	111
		Average	**119**	**122**	**116**
Heavy	Heavy	1	92	92	88
		2	92	93	88
		Average	**92**	**93**	**88**

Table 8 con't Red, green and blue colour values (0-255) for samples subjected to varying degrees of grit-blasting and Scotchbrite® abrasion.

Scotchbrite	Grit-blast	Position	Red	Green	Blue
Light	None	1	194	200	190
		2	201	203	193
		3	225	235	216
		4	184	179	175
		5	177	172	166
		Average	**196**	**198**	**188**
	Light	1	180	179	175
		2	166	163	160
		3	166	164	160
		4	178	186	178
		5	178	186	171
		Average	**174**	**176**	**169**
	Medium-Light	1	148	153	142
		2	144	145	135
		3	138	144	134
		4	135	134	129
		5	145	146	139
		Average	**142**	**144**	**136**
	Medium	1	121	124	118
		2	119	123	116
		3	125	130	126
		4	120	126	118
		Average	**121**	**126**	**120**
	Heavy	**1**	**83**	**85**	**80**

3.3.2 Gloss-meter measurements for grit-blast severity

Table 9 indicates the range of gloss unit values obtained using the Hand Held Mirror TRI Gloss®. For a heavily abraded surface there is a difference of approximately 7 units from 12 to 5 as the grit-blast approaches a level around "4". There is a difference of approximately 18 units from 23 to 5 as the grit-blast approaches a standard level of "4" for surfaces that are abraded lightly. In absolute percentage terms the gloss units show significantly greater discrimination than the colour meter measurements. Further work at all three angles will be needed to clearly establish the best angle to use for discriminating the levels of abrasion and grit-blasting.

Table 9 Gloss-meter measurements for grit-blast and Scotchbrite® abraded samples subjected to varying degrees of abrasion and grit-blasting.

Scotchbrite	**Grit-blast**	**Gloss Units (mean, std. dev^n.)**		
		20°	**60°**	**85°**
Heavy	None	---	---	12 (0.5)
	Light	---	---	9.9 (0.3)
	Medium-Light	---	---	9.2 (0.2)
	Medium	0.9	2.5	4.9 (0.1)
Light	None	4.2	16.6	22.8 (1.3)
	Light	---	---	15.4 (1.6)
	Medium-Light	---	---	11.6 (0.4)
	Medium	---	---	4.9 (0.2)

3.3.3 Wedge test performance resulting from use of epoxy silane kits

Table 10 indicates the wedge test performance for samples produced using the grit-blast and epoxy silane treatment (sec. 2.1) using the epoxy silane kits described in section 2.4.3. The epoxy silane kits were artificially aged by placing the containers of water, epoxy silane and 0.1% acetic acid in a 50°C oven for periods up to several months. It is apparent that after 3 months little aging has occurred and wedge test results still pass the criteria specified in the engineering standard [2]. Table 11 indicates that the wedge test manufactured at BSTT using the epoxy silane kit performed satisfactorily. Feedback from BSTT included the following comments:

- The silane kit was simple and convenient to use.
- The instructions were clear and easy to understand.
- This kit would be a great improvement on the current method for mixing silane, as everything required is in the kit.
- There is less chance of introducing contamination as there is no need to clean beakers and measuring equipment. As this kit does not require a magnetic stirrer, it would simplify use out in the field or on flight line.
- The inclusion of MSDS was good as it emphasises the importance of correct PPE and handling procedures.
- When receiving the kit it was found that the packaging was more than adequate.
- When the kit was trialed it was found that container "A" was difficult to use. This was due to the size of its mouth and the need for the technician to repeatedly dip aerospace wipes into the container without touching the edge. As the level of silane mix decreased the user had to reach further down with the wipe. Without a lot of care the wipe or gloves could touch the edge of the container. It is suggested that the dimensions of container "A" be changed to something that was shorter and wider with a larger opening. This

would make the kit much easier to use. It is timely at this point to explain that BSTT do not use paint brushes to apply silane as they are hard to check for cleanliness and continued dipping of the brush encourages cross contamination. BSTT prefers to use, and teaches others to use, folded aerospace wipes rather than a paint brush. With this technique a wipe is folded to be approximately 50 mm wide and 120 mm long, dipped into the silane solution and used in a brushing motion to wet the prepared surface. When more silane is needed the wet aerospace wipe is thrown away (no double dipping, this reduces contamination) and a new wipe is folded and dipped.

- When shaking the mix it was noted that there was not enough air gap in the container. This reduced the effectiveness of mixing by shaking. If container "A" was slightly larger (600ml) this would give more room for the mixture to move when being shaken.

From the BSTT comments, it is clear that alternative containers for storing the water are required, however, the current trials can still continue, as the material they are manufactured from would remain the same and, therefore, maintain the relevance of the results. It should also be noted that Nalgene's testing of their containers for chemical resistance is typically performed for 30 days at 50^{o}C.

Table 10 Wedge test performance for FM-300 samples bonded to Al-2024T3 clad alloy pretreated using epoxy silane kits aged at 50^{o}C over a period of months.

Aging time at 50^{o}C (days)	**Specimen**	**Crack at 0 hours (mm)**	**Crack at 24 hours (mm)**	**Crack at 48 hours (mm)**	**Cohesive Failure Area (%)**
0	1	39.0	42.0	42.0	100
	2	38.3	40.0	40.0	100
	3	40.0	40.0	40.0	100
	4	37.0	39.0	39.0	100
	5	37.0	39.0	39.0	100
	Average	**38.3**	**40.0**	**40.0**	**100**
7	1	37.0	39.2	39.2	100
	2	36.2	38.2	38.5	100
	3	35.2	36.5	36.9	100
	4	37.7	39.5	39.8	100
	5	36.3	37.9	37.9	100
	Average	**36.5**	**38.3**	**38.5**	**100**

Table 10 con't Wedge test performance for FM-300 samples bonded to Al-2024T3 clad alloy pretreated using epoxy silane kits aged at 50°C over a period of months.

Aging time at 50°C (days)	Specimen	Crack at 0 hours (mm)	Crack at 24 hours (mm)	Crack at 48 hours (mm)	Cohesive Failure Area (%)
30	1	37.9	39.5	39.5	100
	2	40.9	42.9	43.7	100
	3	38.1	40.2	40.2	100
	4	39.9	42.0	42.0	100
	5	40.0	42.4	42.9	98
	Average	**39.4**	**41.4**	**41.7**	**99.6**
90	1	39.1	40.4	40.4	100
	2	37.0	38.3	39.0	100
	3	36.1	36.1	36.7	100
	4	36.5	38.3	38.3	100
	5	41.5	43.0	43.6	100
	Average	**38.0**	**39.2**	**39.6**	**100**

Table 11 BSTT wedge test result for epoxy silane kit shipped to Amberley for trial

Aging time at 50°C (days)	Specimen	Crack at 0 hours (mm)	Crack at 24 hours (mm)	Crack at 48 hours (mm)	Cohesive Failure Area (%)
0	1	41.0	41.3	41.3	100
	2	37.4	38.3	38.6	100
	3	39.4	40.7	40.7	100
	4	40.4	41.1	41.1	100
	5	39.9	41.6	42.8	100
	Average	**39.6**	**40.6**	**40.9**	**100**

3.4 BSTT bonding facility site visit by DSTO

3.4.1 Bonding facilities

The training facility bond shop is well laid and compact. Entry is into a training area which has two work benches and roll racks. The area appears to be used predominantly for metal bonding training such as honeycomb replacement and metal to metal skin repairs. The room also serves the function of an air lock for the true clean rooms.

Upon entering, the first room to the right is an office space, which doubles as a curing area. Novatech HBC 43 Hot Bonders are used for this; two are set up in the corner of the room. Adjacent to the Office/Curing Room area is a Lay-Up room containing the grit-blaster, curing ovens, epoxy silane coupling agent, lay-up table & PPE lockers. The Lay-Up room can be considered the first of the clean rooms. Temperature and humidity control and monitoring provides a suitable environment for adhesive thawing and application to the bonding substrate.

The last room of interest is the Solvent Cleaning room, accessed though the Lay-Up room. Both Lay-Up and Solvent Cleaning rooms have the air-conditioning interconnected, which potentially may effect temperature and humidity control, particularly in the Lay-Up room where moisture can lead to voiding during vacuum cure (refer section 3.4.2). The Solvent Cleaning room contains two Laminar Flow benches; a safety shower and solvent squeeze bottles (MEK) and is used to surface prepare the aluminium substrates.

3.4.2 Environment

As the wedge test specimens were surface prepared in the Solvent Cleaning (SC) room and were assembled in the Lay-Up (LU) room, the investigation was centred on these two rooms. On the day of the visit the rooms were clean and tidy and it was obvious the instructors had a good housekeeping regime in place. Nobody was permitted entry without putting on fresh disposable overalls and personal protective equipment (PPE), which was mandatory. The personnel who required solvents used butyl rubber gloves to protect themselves and surgical gloves over the butyl rubber gloves to protect the wedge test substrates from contamination; the surgical gloves were worn for a maximum of five minutes. The laminar flow benches effectively removed the solvent fumes, although the make-up air was not conditioned

The rooms in question were strictly used for training and re-qualification purposes and were generally not used for production. During the time of the visit an oven in the lay-up room was used to cure a production component due to problems with the production oven. The lay-up table was covered with fresh teflon coated glass and this was changed at regular intervals or when contamination was suspected. In addition to the teflon coated glass, all working surfaces were covered with fresh brown kraft paper to prevent cross contamination. Brown kraft paper was also placed under the wedge test substrates during the surface preparation stage; this was changed at each step and whenever the trainee suspected contamination.

The LU and SC rooms had an interconnected air conditioning system and on the day of the visit the rooms were 23°C and the relative humidity 59.9%, as measured by equipment located on a wall of the LU room. This was marginally over the recommended range prescribed in DEF(AUST)9005 Issue A [1]. An explanation suggested that the make-up air for the laminar flow benches put the room out of specification. The numbers observed were not a cause for great concern but prolonged use of the laminar flow benches may cause problems.

The consumables were stored out of the way on a rack in the metal to metal repair area and it is recommended that the addition of a dust cover would be appropriate. The contents of the bins were examined with only two inappropriate items being found; a milk carton in the metal to metal repair area and food wrappers in the curing/office space. This highlighted the problem of having offices effectively inside a bond shop. Food should not be permitted in the bond shop for obvious reasons.

Generally, the quality and maintenance of the bonding facilities were considered to be of a very high standard and there were no obvious indications of deviations from prescribed standards nor clear cases that may be linked to the deterioration in bond quality observed with the wedge testing.

3.4.3 Sample collection

A range of samples were collected from the bonding facilities, mainly from the LU and SC rooms where the critical surface cleaning and bonding operations occured. The samples included 1) the brown kraft paper used as a barrier layer on most benches and for wedge test preparation, (2) swabs from benches, beakers and general equipment used in bond preparation and (3) samples of Scotchbrite®, tissues and surgical gloves. A sample of the epoxy silane was also received from BSTT prior to the visit and analysed by infrared spectroscopy as detailed in section 3.5. Infrared analysis of items collected from BSTT was initially restricted to the brown kraft paper used in bonding operations, due to evidence from BSTT staff that the supplier for this item had changed during the period that wedge test results appeared to have deteriorated. FT-IR microscopy[5] of the brown kraft paper suggested small differences in the chemistry, however, both samples were essentially cellulose with silica incorporated in the shiny side of the paper. There was no evidence of silicone type contamination for the paper.

3.4.4 Wedge test manufacture and observations of BSTT and DSTO bonding procedures

DSTO and BSTT technicians both prepared wedge tests in the BSTT bonding facility on 13/11/03. Subsequent testing of the specimens at DSTO revealed both passed, with no adhesion[1] failure observed in the crack-growth region and crack-growth being far below the levels defined in the RAAF standard [2]. Results are detailed in Table 12. BSTT wedge failure surfaces revealed much lower voiding levels of the sub-millimetre dimension compared to DSTO. DSTO's wedges also had large voided areas on samples 2, 3 and 4 (Figure 5). XPS analysis of the large voided area is shown in Table 13. The two surfaces look to be similar in composition and there was no silicon or aluminium detected, suggesting that the metallic side was covered in epoxy adhesive. This suggests that the voided regions on the metallic sides were not a result of inadequate

[5] Refer to the Glossary for a definition of IR microscopy.

surface treatment. It is possible the failure mode created by the peel angle induced during separation of the wedges post failure may be in some way responsible. Typically, samples that fail at high peel angles show apparent adhesion failure, however, more detailed failure analysis usually shows that the crack propagates in close proximity to the adhesive-metal interface, but within the adhesive layer [7]. A review of the method used to separate the adherends by the technician revealed that in some instances very high peel angles were created. The procedure to separate adherends has now been modified to avoid these high angles and subsequently the large "voided" regions are no longer being observed on the separated wedge surfaces. The colour variation in the outer samples (Figure 5) may reflect reduced thickness and extra voiding due to the uneven pressure provided by the vacuum bag curing process.

Table 12 Results for wedge tests manufactured at BSTT by technicians from DSTO and BSTT.

Technician	**Specimen**	**Crack at 0 hours (mm)**	**Crack at 24 hours (mm)**	**Crack at 48 hours (mm)**	**Cohesive Failure Area (%)**
DSTO		40.6	42.7	42.7	100
		40.1	40.1	40.5	100
		39.9	41.0	41.0	100
		40.9	40.9	41.6	100
		43.5	45.6	45.9	100
	Average	**41.0**	**42.06**	**42.3**	**100**
BSTT		39.7	41.0	41.0	100
		38.6	39.6	39.6	100
		38.1	39.0	39.3	100
		38.9	39.5	40.4	100
		42.8	43.6	43.6	100
	Average	**39.6**	**40.5**	**40.78**	**100**

Table 13 XPS analysis of voided region on sample 4 of DSTO's wedge test

Side	**%C**	**%O**	**%N**	**%Br**
"metal"	93.9	4.7	0.9	0.6
"adhesive"	91.8	6.0	1.4	0.8

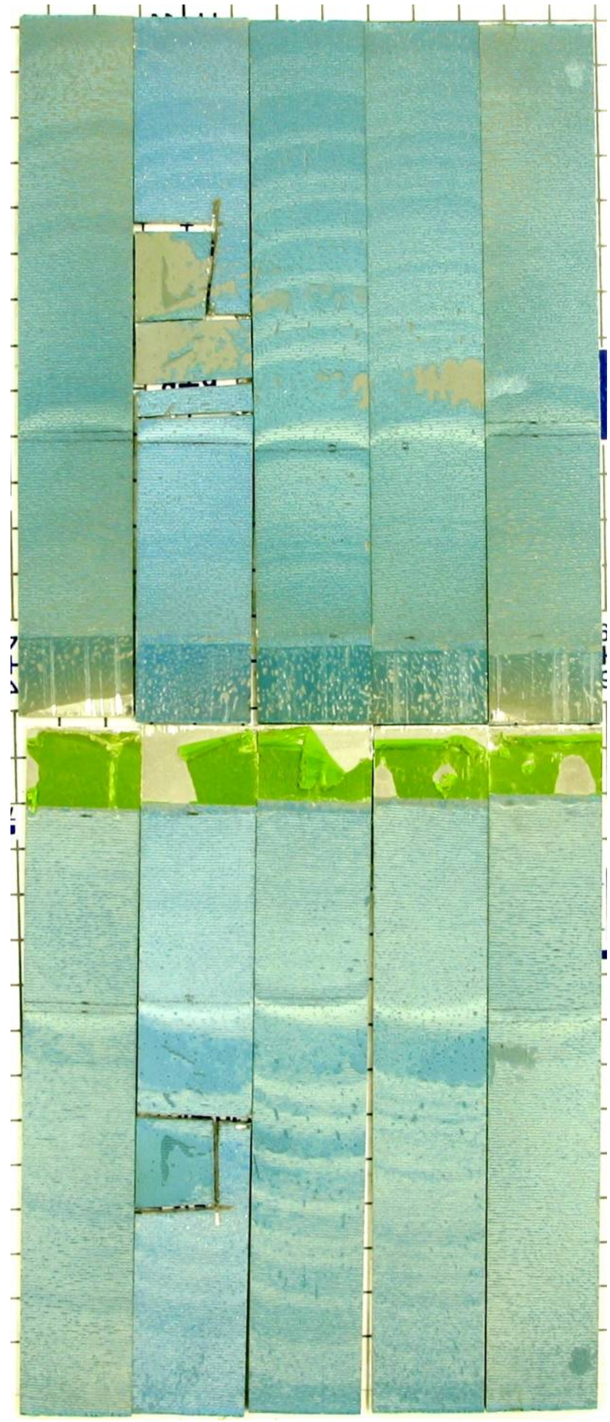

Figure 5 *Images of DSTO's wedge samples after testing and separation. Samples are numbered from 1 to 5 from right to left. Samples 2, 3 and particularly 4 show large voided areas. XPS analysis of the voided region in 4 was performed on the samples produced from the cut regions.*

During wedge test manufacture and observation of technicians being qualified by the BSTT examiners, the processes being implemented by DSTO and BSTT were observed. It should be noted that the differences do not imply deficiencies in either of the methods applied, rather changes that occur in processes over time due to a wide range of factors. The major points observed for the BSTT and DSTO wedge preparations included the following:

1) BSTT staff may employ a wipe cloth to the epoxy silane wetted surface to remove excess solution and facilitate the even drying of the surface
2) BSTT supervision is intense and any deviations from the prescribed methods are noted and the technician would be failed if any step isn't done exactly as specified
3) BSTT staff are strictly implementing the Engineering standard for training and qualification and to the highest quality
4) BSTT trained technicians will apply the silane with wipe cloths soaked in solution compared to DSTO technicians who employ cleaned paint brushes for the same process.
5) The standard of technicians being qualified on the days during the visit was of the highest order
6) BSTT trained technicians use far less pressure and time in the Scotchbrite abrasion steps compared with DSTO
7) BSTT trained technicians prepare the wedge test in a faster time than DSTO technicians
8) BSTT use vacuum bag pressure and heater blankets for adhesive curing of the wedge test plates, compared with positive pressure from a platen press at DSTO.

3.5 Infrared analysis of epoxy silane (γ-GPS)

Appendix A includes the RAIR spectra of the epoxy silane (γ-GPS) acquired from BSTT Amberley and Sigma-Aldrich (Figure A8). The spectra show that the peak intensity of both samples at 1105 cm^{-1} (due to Si-O-Si vibration in the cross-linked film) is about 0.005 absorbance units, indicating a very thin film of approximately 3nm thickness. In contrast, the spectrum acquired for the film produced by the aged γ-GPS (Figure A9) shows a peak intensity of 0.12 absorbance units and clearly substantially thicker than the films produced by the fresh γ-GPS. The aged γ-GPS film also shows a substantial peak due to carbonyl vibration (1731cm^{-1}) and the peak at 825cm^{-1} is due to methoxy species present from unhydrolysed γ-GPS. The transmission infrared spectra of the fresh and aged epoxy silanes in Figure A10 clearly indicate differences in the peaks at 3513 and 1730cm^{-1}, due to hydroxyl and carbonyl vibrations, respectively. The aged silane shows that carbonyl and hydroxyl impurities increase substantially and show that both the transmission and RAIR spectra can be used to identify epoxy silane age and purity.

3.6 Wedge test sensitivity studies

Table 14 indicates wedge test results for the basic grit-blast and epoxy silane treatment for examples where critical changes are made to the process or process environment. It can be seen that the absence of grit-blasting leads to a greater variation in final crack growth values at 48 hours. There is also a clear increase in the average crack length value at 24 and 48 hours and the failure mode becomes 100% adhesion. Very similar trends are observed for the wedge manufactured in the silicone environment, representing the influence of surface contaminant. The increase in crack length and large adhesion failure observed for the samples that were not grit-blasted are very similar to values observed for the wedge test specimens that failed in Table 2 and were identified as having inadequately grit-blasted surfaces.

A disturbing aspect of the wedge test manufactured in the silicone room environment was the lack of sensitivity of the water break test currently being used to identify the presence of surface contaminant. Given the clear reduction in quality of the wedge test results, silicone contaminant was obviously present. This indicates improvement in the methods to identify surface contamination are required if the wedge test results are to become more reliable.

Not surprisingly, the absence of epoxy silane in the treatment step results in the largest crack-growth observed for all treatment variations and 100% adhesion failure, clearly indicating the importance of correct application of the epoxy silane. The final average crack length is similar to the worst cases observed for the BSTT wedge qualification samples (Table 2).

The absence of abrasion or careful detritus removal after abrasion, unexpectedly, had no influence on wedge test performance with both these tests providing acceptable crack growth and 100% cohesion failure. This result indicates the grit-blasting step is crucial to overall performance of the treatment. Possibly, grit-blasting consolidates the surface debris and reduces the potential of a weak boundary layer forming.

Table 14 Wedge test sensitivity studies examining the influence on crack growth and failure mode in cases where critical changes are made to the bonding process.

Treatment	Specimen	Crack at 0 hours (mm)	Crack at 24 hours (mm)	Crack at 48 hours (mm)	Cohesive Failure Area (%)
Standard grit-blast and epoxy silane process (sec. 2.1)	1	39.0	42.0	42.0	100
	2	38.3	40.0	40.0	100
	3	40.0	40.0	40.0	100
	4	37.0	39.0	39.0	100
	5	37.0	39.0	39.0	100
	Average	**38.3**	**40.0**	**40.0**	**100**
No grit-blasting (step 6 sec. 2.1)	1	39.8	45.4	48.1	0
	2	37.5	47.3	47.3	0
	3	34.4	36.5	42.1	0
	4	34.4	37.0	37.1	0
	5	39.8	48.0	48.0	0
	Average	**37.2**	**42.8**	**44.5**	**0**
Standard process (sec. 2.1) - in silicone room	1	36.5	41.8	44.8	0
	2	35.0	43.8	43.8	0
	3	36.0	41.3	43.3	0
	4	36.0	43.8	47.3	0
	5	36.5	41.8	43.3	0
	Average	**36.0**	**42.5**	**44.5**	**0**
Standard process (sec. 2.1) -no abrasion	1	36.1	37.9	37.9	100
	2	34.6	36.3	36.3	100
	3	35.9	37.8	37.8	100
	4	34.1	35.7	35.7	100
	5	37.1	39.0	39.0	100
	Average	35.6	37.3	37.3	100
Standard process (sec. 2.1) -no detritus removal	1	37.2	39.5	40.2	100
	2	33.5	36.1	36.1	100
	3	36.6	37.6	38.8	100
	4	33.7	36.0	37.6	100
	5	37.5	39.3	40.2	100
	Average	**35.7**	**37.7**	**38.6**	**100**

Table 14 con't Wedge test sensitivity studies examining the influence on crack growth and failure mode in cases where critical changes are made to the bonding process.

Treatment	Specimen	Crack at 0 hours (mm)	Crack at 24 hours (mm)	Crack at 48 hours (mm)	Cohesive Failure Area (%)
Standard process (sec. 2.1) -no epoxy silane	1	38.3	45.9	50.6	0
	2	36.4	43.8	50.3	0
	3	39.3	46.1	51.3	0
	4	38.3	43.8	49.5	0
	5	38.1	46.9	52.5	0
	Average	**38.1**	**45.3**	**50.8**	**0**
-abrasion only	1	40.31	59.48	60.7	0
	2	36.23	62.68	63.18	0
	3	36.82	63.00	63.81	0
	4	35.86	62.15	62.76	0
	5	40.4	60.94	61.70	0
	Average	**37.9**	**61.7**	**62.4**	**0**
-solvent degrease only	1	46.26	80.05	80.05	0
	2	35.76	82.61	82.61	0
	3	39.84	84.30	84.30	0
	4	48.28	85.96	85.96	0
	5	52.39	81.80	81.80	0
	Average	**44.5**	**82.94**	**82.94**	**0**

4. Discussion

4.1 BSTT Qualification- wedge test inspection

4.1.1 Failure rates and effects of grit-blast and adhesive age

From the results in Table 2 it is clear that FM-300-2K cannot be used in wedge test requalification using the present system. The failure rate of almost 65% is five times greater than the rate observed for FM-300. Of the 14 failed samples resulting from wedge testing using FM-300, 50% of these exhibited levels of grit-blasting that were visually insufficient (Table 3), and were at levels of 2 or less, as indicated in Figure 3. The reason for the high failure rate for FM-300-2K is not obvious, but the adhesive bond durability is apparently far more sensitive to variations in the processes and materials used in the wedge test manufacturing. As indicated in the Glossary, FM-300 cures at 177°C for 60 minutes, whereas FM-300-2K cures for 90 minutes at 120°C. It may be that the lower curing temperature reduces the ability of FM-300-2K to displace contaminants present on the metal surface and is, therefore, more sensitive to variations in surface cleanliness.

If it was assumed that the inadequate grit-blasting observed in 50% of the failed FM-300 samples could have been prevented through implementation of surface roughness measurements using tools described in section 2.4.1, then the overall failure rate for the FM-300 samples would be reduced from 13% to 6.5%. Group 14 was responsible for 50% of all failures recorded for FM-300, however, only 2 of those samples exhibited inadequate grit-blasting, leaving 70% of the failed samples for this group unexplained. The only obvious differences between group 14 and other groups was the Edinburgh test location in South Australia and the first shipment of FM-300 sent to the test location from Amberley may have been exposed to extended storage at room temperature. Despite the concern with the first adhesive shipment, failures occurred at similar rates for the first and second shipments and on adequately grit-blasted surfaces.

Potential explanations for failure not related to inadequate grit-blasting may include the period between training and requalification or adhesive aging. Table 3 indicates that more failure for FM-300 occurred in requalification than directly after training. This may suggest that if personnel are not regularly involved in bonding operations various aspects of the wedge test manufacture that are not directly examined during requalification testing may be overlooked or forgotten. The high failure rate in group 14, however, distorts the data and more information would be needed to support this case. Boeing-Amberley, however, has a relatively higher rate of success with only 1 failure in 38 not being explained. A large number of bonded repairs to F-111 honeycomb panels are performed by Boeing Amberley technicians. The higher RAAF failure rate includes results from over 15 different units and it may be expected regular involvement in bonding would not occur for all those involved in requalification. The conclusion that regular practical application of bonding skills may be needed is an indication that the current process applied by RAAF may have limitations. It may require improvements to increase the failure rate to acceptable levels, if the wedge test result was to be used as the sole criterion for qualification of bonding technicians.

The adhesive age as a function of failure rate shown in Figure 4 suggests no obvious trends. In contrast, the similar failure rates observed for the first two batches of adhesive used may suggest that the failure rates are a result of the process being applied and would imply inadequacies in the present controls being implemented.

Another variable that should be considered is the adhesive batch being used. Figure 6 indicates that the average crack length after 48 hours has increased with respect to the mean crack growth for the three adhesive batches used. This trend either indicates deterioration in the adhesive being supplied from the manufacturer or a gradual decline in the quality of the adhesive bonding processes.

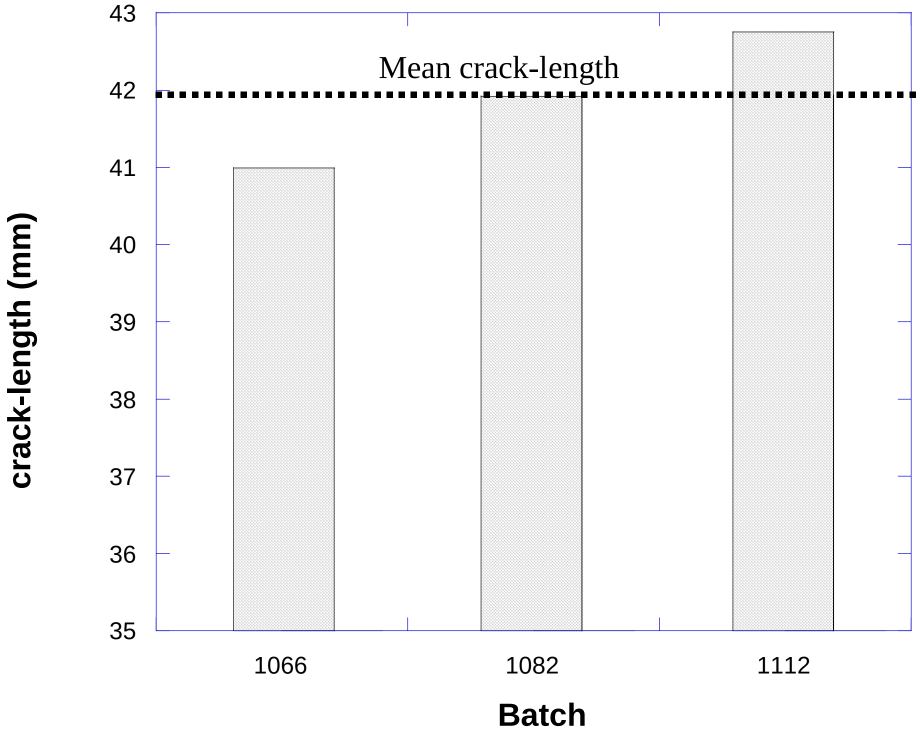

Figure 6   48 hour crack length for FM-300 for the three batches used compared with the mean crack growth for all specimens tested.

4.1.2 Failure analysis

XPS analysis of the specimens from groups 11, 14 and 20 (Table 7) reveal that no silicon is detected on the metallic failure surface for any samples. There is evidence from the results in Table 7 that as the final crack length after 48 hours of humid exposure increases (i.e. the bond becomes less durable) the crack propagates closer to the adhesive and aluminium interface. The absence of silicon would suggest that the epoxy silane coupling agent is not retained on the metallic surface and the hydrolytic stability of the interfacial region deteriorates as the bond becomes less durable. It is possible that some variation in the current manufacturing process that is not being monitored has the potential to reduce the bond stability between the epoxy silane and grit-blasted aluminium surface in a humid environment.

4.1.3 Quality control

Examination of the Colour and Gloss meter units in sections 3.3.1 and 3.3.2 clearly indicate commercially available units are capable of discriminating between different levels of grit-blasting on clad Al-2024 T3 surfaces. Whilst the Gloss meter is significantly more costly, the improved discrimination and data down-loading capacity would make it the preferred candidate, particularly, when the wedge sensitivity studies (section 3.6) confirm the critical nature of the grit-blasting step in the overall bonding process.

Trialing of epoxy silane kits has proven to be successful to date (section 2.4.3). The ability to package epoxy silane, water and acetic acid in ready-to-mix kits offers advantages in transport, traceability and quality control. Small improvements to the prototype can be made by altering the dimensions and volume of water stored in the largest container and by adding the diluted acetic acid to the water during packaging. The aging studies to date have shown no deterioration in wedge test results for 90 days in 50^{o}C, which is three times longer than the maximum time used by the container manufacturer in assessing their chemical resistance.

Development of infrared spectroscopic techniques (section 2.4.2) has now provided a method of qualifying the epoxy silane on a batch-to-batch basis and enabling identification of changes that may be detrimental to the bonding operation. The technique developed uses standard infrared equipment available in most analytical laboratories and surface treatments that are within the capability of basic metallurgical facilities. In combination with the epoxy silane kits there will now be a capability to trace the quality of the batch that has been used in any requalification or bonded repair operation by RAAF.

The water break test has indicated some limitations in identifying silicone contaminant on the prepared aluminium surface at levels that can degrade the adhesive bond durability. Alternative and more reliable quality control tools are required to analyse the bonding surface. Presently, infrared spectroscopic techniques have potential in this area [8], however, their application relies to some extent on the skill of the operator employing the technique. The equipment used to measure the infrared spectrum of a metallic surface is also generally restricted to laboratory environments and modifications to standard equipment would be required to adapt the process to field or production environments similar to those experienced at BSTT, Amberley [8]. Work by Dr David Arnott and co-workers at DSTO several years ago identified a unit called the SQM-200 for measuring surface contaminant [9]. The unit works on the basis of shining ultraviolet light at a metal surface and detecting the electrons stimulated by the radiation. The flux of optically stimulated electrons relies on the metal surface's work function and is affected by monolayer levels of contaminant. Previous research has indicated the high sensitivity of the equipment and it would be sensible to attempt to implement it for quality control monitoring in wedge test fabrication.

4.1.4 BSTT bonding facility site visit

Inspection of the BSTT bonding facility by DSTO indicated very high standards are being maintained. Minor transgressions were observed, however, these were not considered to be significant in terms of the problem that had developed with wedge test quality. Some concerns were raised about humidity and temperature control being slightly outside limits and is an issue that would need to be considered, particularly when using a vacuum bag arrangement to cure the wedge test specimens. Previous work at DSTO has highlighted the effect that high humidity levels can have on void development in the adhesive during cure [10]. Despite a range of possible materials potentially being the cause for problems in bonding, only the epoxy silane and the brown "kraft" paper could be identified as having changed supplier or batch during the period when bonding began to deteriorate. As detailed in section 3.5, analysis of the BSTT epoxy silane showed no significant variation with the fresh batch received from Aldrich and indicated this wasn't a likely source of the problem. Infrared analysis also indicated that little variation in the paper had occurred and would not have contributed to the deteriorating bonding performance.

Wedge tests manufactured by BSTT and DSTO during the DSTO visit, both passed the criteria defined in the RAAF standard, however, differences in the processes were observed. Generally, BSTT was faster at completing the surface preparation tasks, which was mainly the result of reduced time on the abrasion steps and more efficient detritus wiping. The wedge test sensitivity studies (Table 14) suggest that reduced abrasion is not likely to be a significant factor affecting bond durability, however, in practical bonding situations where thick oxide and contaminant layers may be present the step is still highly important. Whilst the sensitivity studies provide a good indication of possible variations, additional testing is required to establish a reliable indication of result variation. Variations in process between the two organisations also involved the application and drying of the epoxy silane. BSTT preferred to employ aerospace wipes to apply the silane as well as remove excess moisture from the surface to prevent pooling of solution during drying. DSTO typically employ cleaned brushes and use these to remove excess solution prior to drying. In either case it was clear that the RAAF standard failed to provide detailed instructions on the preferred drying procedures, although the use of aerospace wipes to apply the silane solution is now utilised by BSTT as it avoids contamination issues involved with cleaning paint brushes. It is unlikely that the variations observed were likely to reduce the durability of adhesive bonds prepared with the grit-blast and silane method.

Measurement of the pH of the 1% aqueous silane solution produced at BSTT indicated a value around 7, compared with 4-5 for solutions prepared at DSTO. The neutral pH value would increase the time required for hydrolysis of the epoxy silane molecule [11] Hydrolysis of the silane produces reactive silanol groups, which bond directly with the metallic surface, and is a critical step in the silane film formation process. At a pH of 4-5 the hydrolysis process is optimised and the minimum of 1 hour solution stirring is adequate to insure hydrolysis has proceeded. At a pH of 7, 1 hour may be insufficient time to achieve the required level of hydrolysis. The implementation of silane kits with the acetic acid will ensure the acidic conditions for accelerated hydrolysis are achieved and should help to minimise variation in the silane film quality.

The only other obvious variation between BSTT and DSTO is the curing procedure. DSTO uses a positive pressure platen press, whereas BSTT employ a vacuum bag arrangement. As referred to above, the vacuum bag set-up will be far more sensitive to humidity and moisture levels on the surface and the adhesive and will be more prone to voiding during cure. Potentially, concerns over voiding have influenced BSTT procedures and the manner in which abrasion and grit-blasting is conducted. By ensuring that the surface is not over grit-blasted and lightly abraded the capacity to trap volatiles in the bondline can be reduced [10]. Unfortunately, light grit-blasting, as is demonstrated in Table 3 can lead to notable deterioration in bond durability. Interestingly, the large voiding and micro-voiding observed for the DSTO wedge sample prepared at Amberley was not observed for the BSTT produced sample (Figure 5). As indicated in section 3.4.4, investigation of the process to separate the wedge test samples after testing revealed that high peeling angles were responsible for inducing peel failure modes close to the adhesive and metal interface. These high peel angles were responsible for the apparent large voids in the DSTO sample. The increased level of micro-voiding in the DSTO sample may suggest that greater abrasion contributes to the retention of moisture. Reducing voiding by altering severity of abrasion and grit-blasting is fraught with danger, however, and every effort to minimise moisture retention on the mating surfaces should be attempted through drying procedures as a first attempt. Having said that, it is often a substantial task to minimise adherend moisture levels in the high humidity regions of Northern Australia.

4.1.5 Wedge test sensitivity studies

The wedge test sensitivity studies (Figure 7 and Table 14) provided a range of predictable and unpredictable results. As anticipated, the absence of grit-blasting results in increased crack growth and 100% adhesion failure, supporting the conclusion that inadequate grit-blasting observed for 50% of the BSTT data that used FM-300 adhesive was responsible for wedge tests failing. Surprisingly, however, absence of abrasion or removing the detritus after abrasion had little influence on bond performance, suggesting that a satisfactory grit-blast can minimise the impact of these steps in the bonding process. The presence of silicone contaminant also, whilst degrading bonding and producing 100% adhesion failure in the cracking zone, did not produce the highest crack growth of all samples manufactured. A major concern was the inability of the water-break test to identify the silicone present on the bonding surface at levels that clearly degraded the bond quality. As indicated above, the use of alternative quality control tools to monitor surface contamination should be examined.

Clearly, the absence of epoxy silane had the greatest impact on the bond durability. The crack-length of over 50mm was similar to the worst wedge test results observed from the BSTT requalification data and may imply that effective silane film formation may not have been achieved in these cases. As described in section 4.1.4, the variation in solution pH is a possible contributor to variation in silane film properties. Efforts to control and monitor solution pH should be considered in increasing the reliability of the bonding process.

The two worst results were achieved when silane and grit-blasting was absent from the treatment or when no abrasion or grit-blasting was used. The results clearly indicate that surface roughening is critical to the overall performance and that if the surface isn't adequately prepared the epoxy silane will have no effect. The results will be valuable in defining response parameters for a regression model that will be developed as a part of a risk and reliability approach to certifying adhesive bond durability.

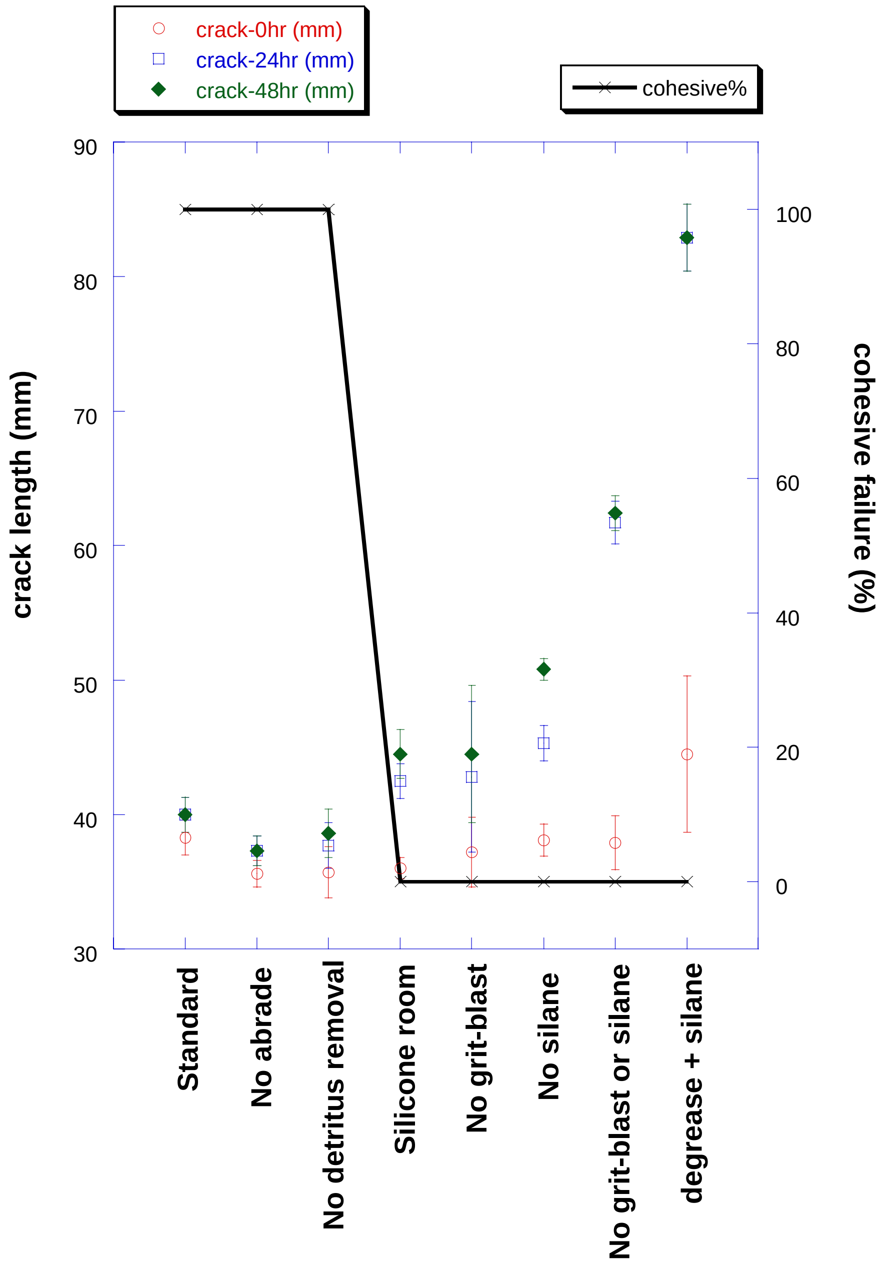

Figure 7   Crack-length as a function of standard grit-blast and epoxy silane process for cases where steps are removed or environment is altered.

5. Conclusions

Examination of 124 wedge test samples (or 620 individual test pieces producing 1240 failure surfaces) and their associated details provided a number of indicators to assist in explaining the wedge test failures observed during qualification testing by BSTT, Amberley.

FM 300-2K cannot be used for qualification testing if the current RAAF standard criteria are used to pass or fail technicians. The adhesive exhibits very high failure rates and is clearly very sensitive to process variations inherent in the grit-blast and epoxy silane treatment currently favoured by RAAF.

Fifty percent of all samples that failed when FM-300 adhesive was used could be directly linked to inadequate grit-blasting. Wedge test sensitivity studies confirmed that inadequate grit-blasting leads to higher crack growth and 100% adhesion failure.

Assessment of commercially available colour and gloss-meter units indicates that the units are capable of discriminating between the different levels of grit-blasting and would be a valuable tool in maintaining quality of the surface treatment process.

Seven of the total fourteen failures using FM-300 occurred for requalification testing conducted at RAAF Base Edinburgh. Despite two cases of poor grit-blasting, the remaining failures could not be readily explained by visual inspection. Surface analysis of failed samples, however, revealed the absence of epoxy silane on the aluminium surface, suggesting there could be some problems with the hydrolytic stability of the deposited film.

Indirect evidence suggests that the experience of the technician and regular application of the skills required in adhesive bonding to metallic structure has an influence on the pass rate for the wedge test. Boeing Amberley technicians exhibited a pass rate almost fifteen percent higher than RAAF technicians. Whilst Boeing technicians are involved in regular bonding operations, RAAF qualification covered fifteen different units where the opportunity to apply the bonding skills learnt through training would vary considerably.

The age and batch of FM-300 adhesive were also examined as factors. The results indicate that whilst failure rates for the two oldest batches were similar, average crack growth measurements have successively increased for each of the three batches employed for qualification testing. There is no clear reason for the gradual increase in the average crack length of wedge samples produced during BSTT testing and requalification.

Review of BSTT bonding procedures and facilities indicated operations were performed to a high level and in strict accordance with the RAAF Engineering

Standard. No obvious sources of contamination that may have contributed to the increase in wedge failure rates were identified, however, particular attention should be paid to ensuring temperature and humidity levels in the bonding facility are maintained well within limits identified in the standard.

Minor variations in the DSTO and BSTT surface treatment methods were observed. The variations included reduced time involved in abrasion and detritus removal, however, wedge sensitivity studies indicated this was unlikely to lead to substantial differences in results.

DSTO and BSTT also employed different methods to apply and remove epoxy silane solution from the metallic surface prior to oven drying. Both techniques can prevent pooling of solution, however, the RAAF standard does not provide detail of the correct process to apply.

Epoxy silane kits that were trialed and received accelerated aging indicated potential in improving the quality of the surface treatment process by minimising contamination sources and errors with mixing. Additionally, the inclusion of acetic acid or some other means to control the solution pH should assist in maintaining the quality of epoxy silane films formed on the metallic substrates.

Wedge sensitivity studies carried out in a silicone room environment showed that the water break test currently employed to monitor surface cleanliness during the surface treatment did not detect contaminant levels that were sufficient to degrade the bond quality. Previous DSTO research suggests that a commercially available unit may be required to improve the quality of the surface monitoring during the bonding process.

6. Recommendations

Based on the findings contained in this report, DSTO recommends the following items be addressed by RAAF in order to improve the quality of the current bonding procedures used for requalification of technicians by BSTT, Amberley :

A quality control tool should be implemented to statistically monitor the wedge test results in real time and highlight deviations in crack-growth from the average trend. This will enable rapid identification of problems with the bonding operation and enable the problems to be solved in a satisfactory time frame.

The wedge test requalification database could be maintained by the Aircraft Forensic Engineering (AFE) group in Air Vehicles Division (AVD), DSTO. This will require BSTT to provide AFE with all the information recorded during the wedge testing examination. Additionally, the technician currently sub-contracted by BSTT through

AVD to test the wedge samples will have to provide AFE with measurement details and specimens.

Commercially available units such as the Hand Held Micro TRI Gloss® gloss-meter from BYK Gardner should be used to monitor grit-blast quality during wedge requalification testing. Some preliminary development work (currently underway) will be required prior to insertion in the BSTT facility.

The SQM-200 from Photoemission Technology should be used to monitor surface cleanliness during surface treatment of the metal surface used for wedge test manufacture. Some preliminary development work (currently underway) will be required prior to insertion in the BSTT facility.

Epoxy Silane kits should be implemented for all bonding operations using the RAAF surface treatment detailed in AAP7021.016-2. This will provide a traceable batch number, improve quality control and provide logistical advantages for BSTT staff when travelling to different bases for requalification testing. Alternative dimensions and volume for the water storage container need to be examined to improve ease of use.

Careful monitoring of adhesive batches throughout their usage life should be considered, including development of spectroscopic or chemical techniques to identify obvious changes in quality.

7. References

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3 IDM Instruments, 6 Carmen Street Dandenong, Victoria, 3175, Australia. Phone 03 9706 7837. www.idminstruments.com.au.

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Appendix A: Infrared analysis of epoxy silane (γ-GPS)

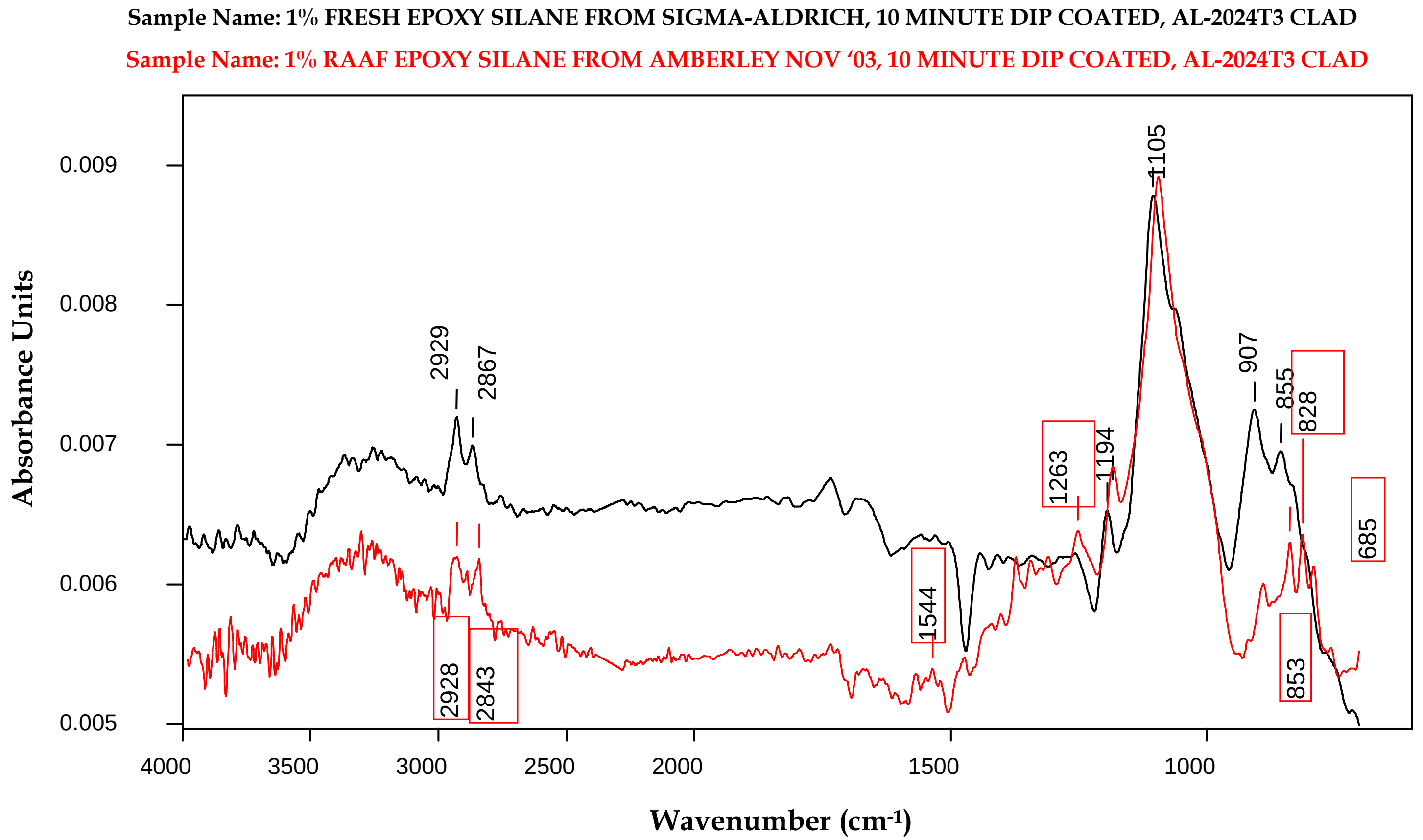


Figure A8 RAIR spectrum of fresh epoxy silane (γ-GPS) from RAAF Amberley and Sigma-Aldrich

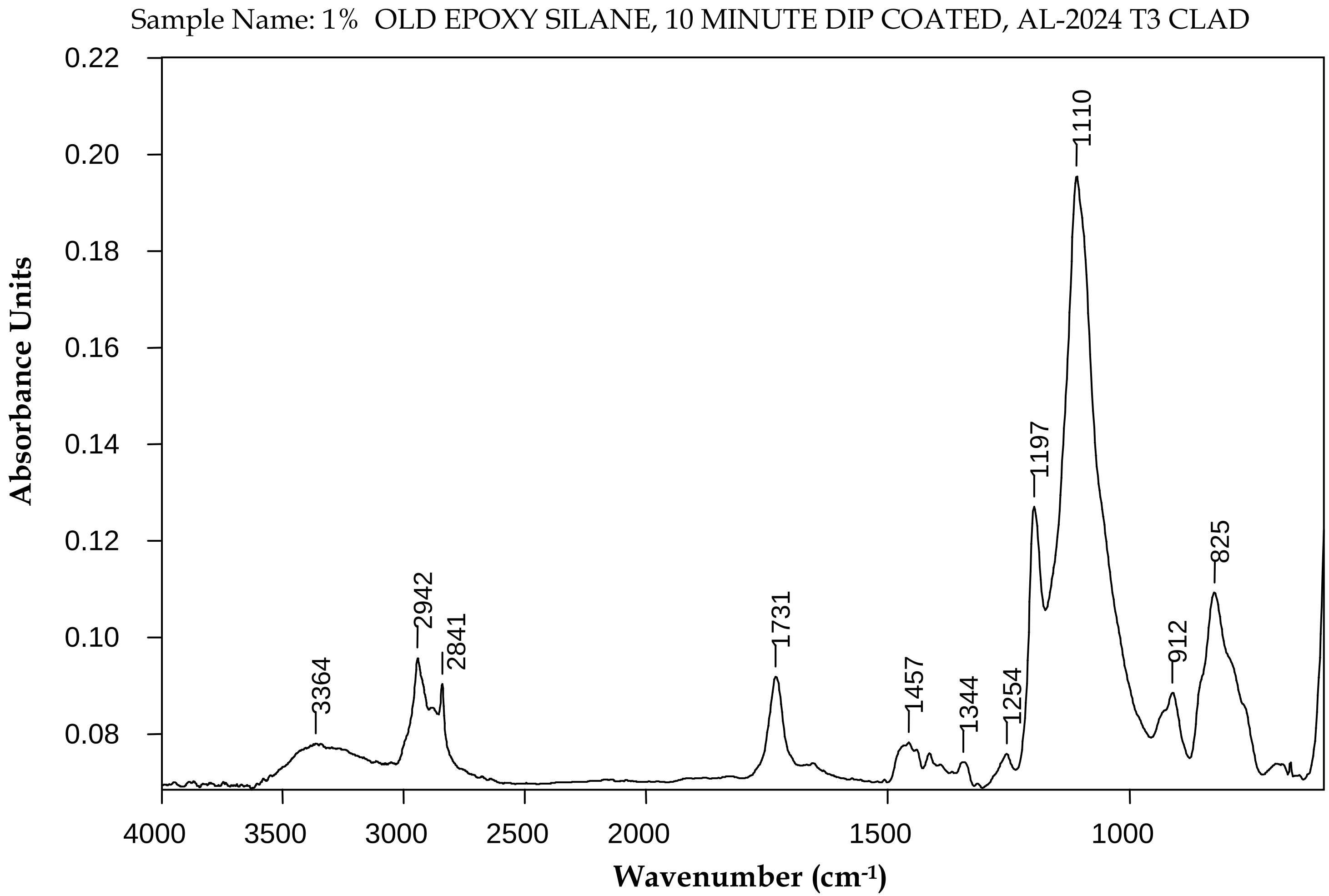
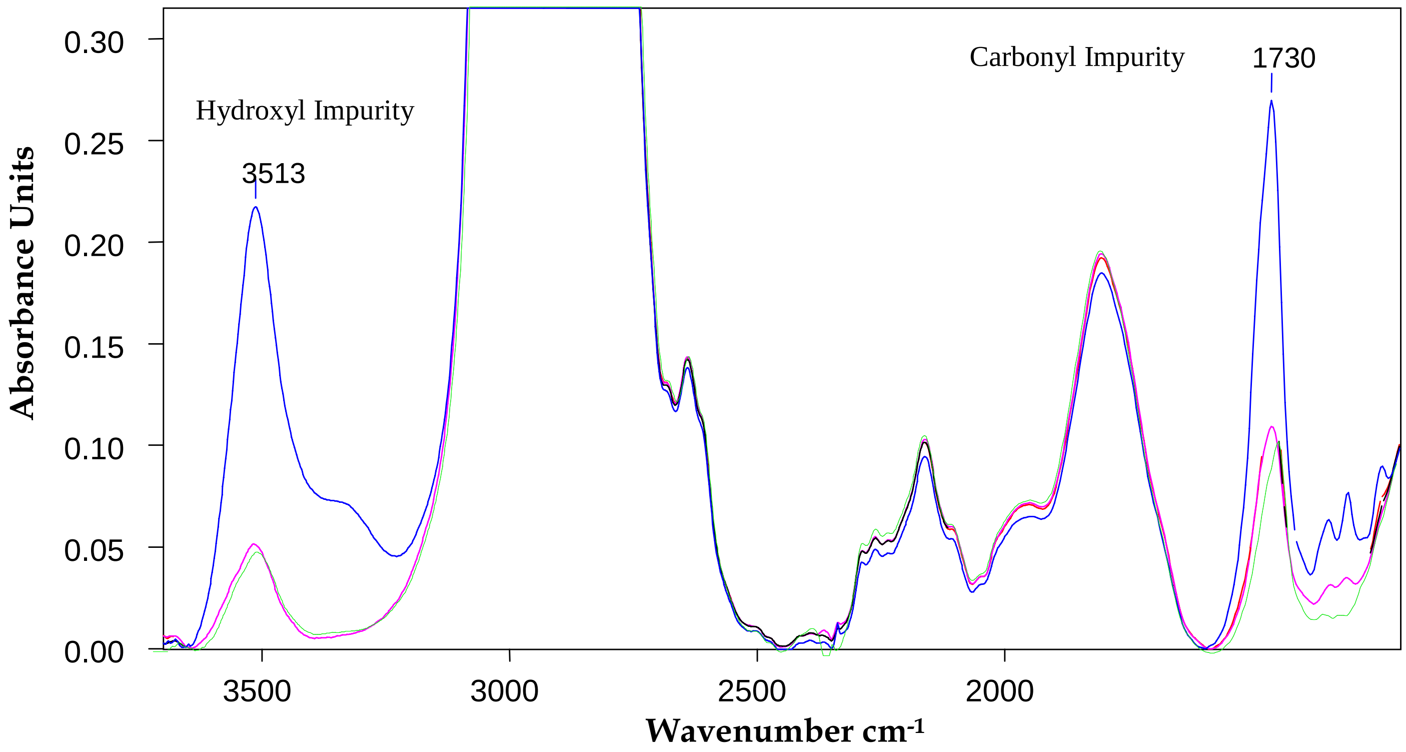


Figure A9 *RAIR spectrum of aged epoxy silane (γ-GPS) from laboratory storage after 6 years.*



Sample Name: OLD EPOXY SILANE AFTER 6 YEARS STORAGE IN LABORATORY.

Sample Name: RAAF EPOXY SILANE FROM AMBERLEY NOV 2003

Sample Name: FRESH EPOXY SILANE, SIGMA-ALDRICH

Figure A10 Transmission Infrared spectra of laboratory aged for 6 years, BSTT Amberley and Sigma-Aldrich epoxy-silane samples

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19. ABSTRACT

DSTO has recently undertaken a review of procedures employed by the Royal Australian Air Force (RAAF) Bonded Structures and Testing Team (BSTT) during testing and requalification of technicians who undertake bonded repairs on ADF aircraft. Currently, technicians are required to produce a bonded wedge test under examination from BSTT staff and, based on adherence to the current RAAF Engineering Standard, will be considered competent to undertake bonded repairs. From December 2001 to January 2004 a notable deterioration in the quality of the bonded wedge tests produced through qualification testing was observed and an audit of processes was undertaken to determine if any areas in the bonding and requalification testing may have been leading to the deterioration in quality. Based on an audit of the processes and subsequent experimental testing at DSTO, recommendations on improvements to bonding procedures have been made.

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